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Horsewell, A.; Hansen, Niels

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Metallurgy Department Annual Progress Report f

Copper, iron and stone sculpture placed in front of the Department.
The artist is Chr. Dahlgaard Larsen.

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Metallurgy Department Annual Progress Report for 1989

Edited by A. Horsewell and N. Hansen

Abstract. Selected activities of the Metallurgy Department at Risø National Laboratory during 1989 are described. The work is presented in three chapters: Materials Science, Materials Engineering and Materials Technology. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

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1. INTRODUCTION

Research Strategy

Research and development are among the key issues of modern society. New research policies and plans are being formulated both in Denmark and within the European Community (EEC). For a research organization like Risø National Laboratory it is vital to be able to adapt to such policies and advise in the formulation of such plans as the Laboratory concentrates on medium and long term strategic research. The main research themes at Risø National Laboratory are energy, environment and materials. In defining and carrying out this research, emphasis is put on the needs of society as well as on the specific demands of the industrial sector. European and international collaboration is seen to be of increasing importance.

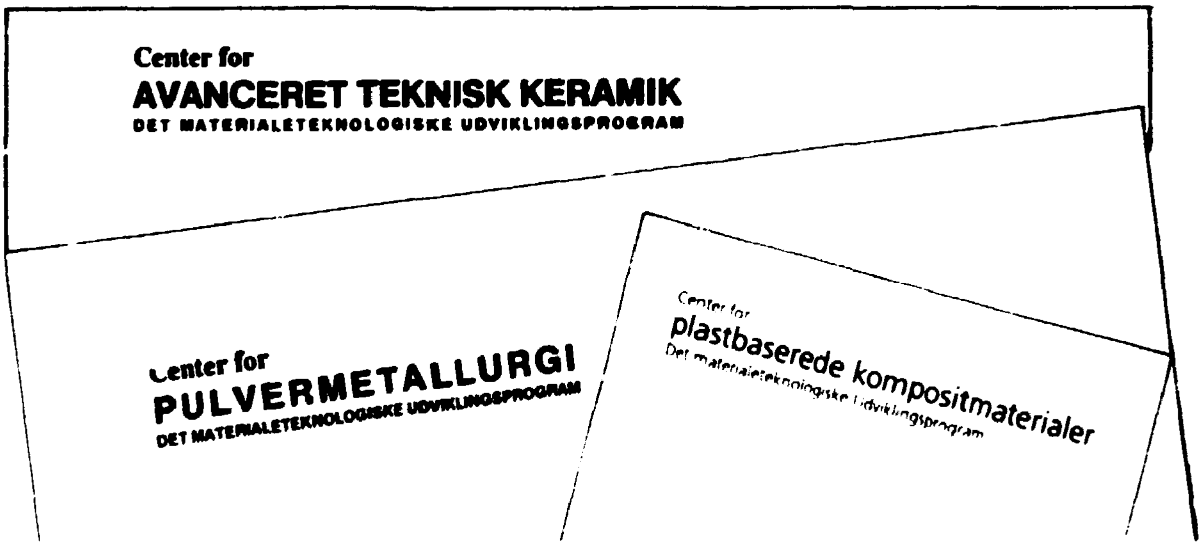
Research and development activities in the Metallurgy Department meet the guidelines mentioned above in such a way that the more applied programmes are based on a longer range effort within more fundamental areas. The fundamental programmes extend our knowledge of materials through, for example, modelling, characterization of microstructure and texture and radiation damage, whereas applied programmes concentrate on areas such as materials properties, engineering, technology and materials performance. The materials groups of interest are metals, polymers and ceramics.

Basic Research

The long range basic research programme covers modelling of materials, behaviour and characterization of structure and properties. The materials investigated are metals, polymers and ceramics. Radiation damage of metallic materials is also studied, especially the problem of simulating the damage which will take place in a fusion reactor. An important part of the basic research programme is the development of advanced experimental techniques within the areas of neutron diffraction (especially texture and internal strain measurement), small angle neutron scattering, positron annihilation and electron microscopy.

Danish Programmes on Materials Technology

Within the general area of materials research the Metallurgy Department is an active partner in the Danish programme on materials technology. This programme, with a budget of DKK 495 mill., is planned for the period 1988 - 1993. About half of this money will be spent on financing five »centres« which are formalized collaborative arrangements between partners from industry, research organizations and universities. The research programme for each centre is agreed upon amongst the partners; the tasks are primarily those given high priority by the industrial partners.



The Metallurgy Department participates in three centres, namely the centre of polymer composites, the centre of powder metallurgy and the centre of advanced technical ceramics. The administration and coordination of two of these centres is placed in the Department.

Research Related to Energy Issues

The materials research specifically in this area is concentrated on problems related to the development of fuel cells. Fuel cells have a high potential for the production of electricity and heat with high efficiency and low atmospheric pollution; hence the high priority given to it by the Ministry of Energy. The work focusses on materials development for solid oxide fuel cells with the aim of constructing a small prototype fuel cell. This research is carried out in close collaboration with the utilities, industry, research institutes and the Ministry of Energy. Energy related research in the Department also encompasses the design and materials development for wind mills which have a high priority within the Danish energy programme. Also within the nuclear fission area, the Third Risø Fission Gas Project has been completed. This marks the end of the Department's conventional nuclear research and as a consequence a large part of the hot cell facility will be decommissioned in the period 1990 - 1993. Finally, in energy related research, the Department participates with materials research within the European fusion technology programme.

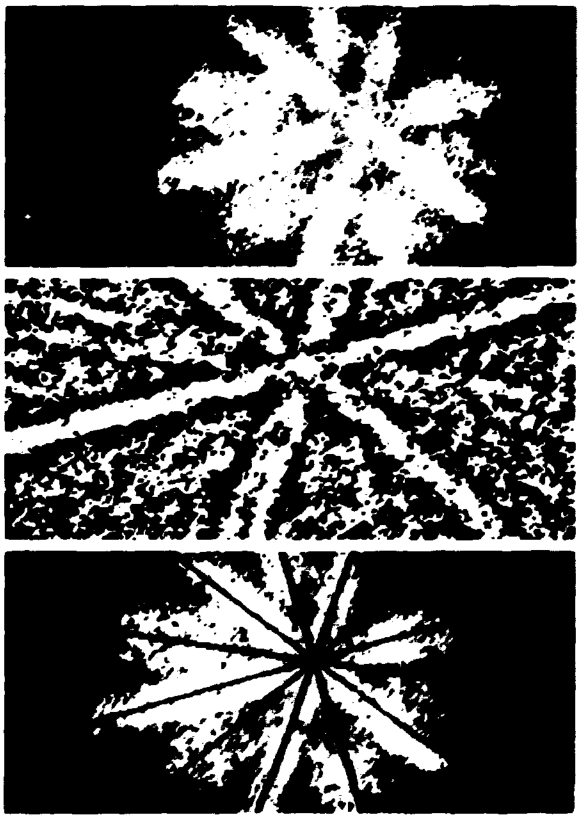
Participation in EEC Funded Research

Participation in Danish research programmes has led to an active engagement in many programmes under the auspices of the EEC. Common ground between Danish and many European programmes has led to partnership within the BRITE/EURAM programme. The Deparment participates in research related to composites, fuel cells, engineering ceramics, brazing and materials testing. Our activities in eight BRITE/EURAM programmes are supplemented by participation in two JOULE programmes on fuel cells and the design and testing of wings for wind mills. The close relationships between the programmes of the Department within the European and the Danish framework supports an important role of the Department as a »bridge« between international and national materials research. This function has an important bearing on issues such as technology import and technology transfer. As part of the European collaboration, staff members have had seats in many committees, advisory groups etc. For details, see Chapter 5.

Research Achievements

A number of scientific and technical achievements made during the year can be mentioned. Within the area of basic research, inhomogeneities in materials have been related to the deformation pattern, to the formation of texture and to the occurrence of mechanical anisotropy; a coherent picture of the relationship between deformation microstructures and properties is emerging. For microstructural studies, the electron back scattering technique has been implemented and the analysis of EBS-patterns has been automatized.

A <111> Electron Back-Scattering Pattern in aluminium. Experimental, computer enhanced and superimposed computer simulation and experimental patterns.



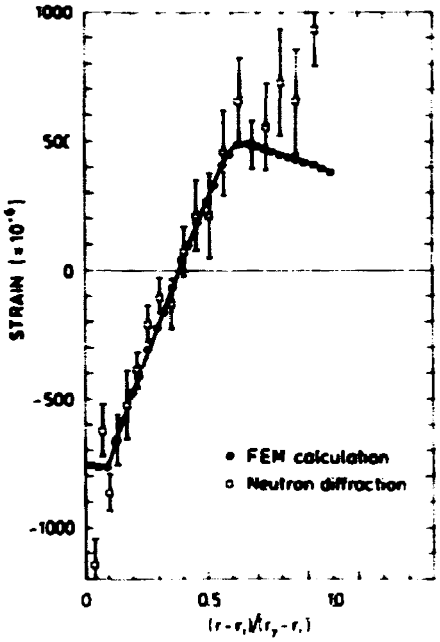
In the area of neutron diffraction measurement of internal elastic strains, a first generation tensile machine for in-situ measurements has been applied to study thermal and mechanical stresses in metal matrix composites. Also within this area, in-depth strain profiles have been determined in thick-walled tubes and near weldments. In the technology field an ultra light polymer composite racing canoe has been designed and built in collaboration with students from the Engineering Academy.

Another technological achievement is the running-in of a new fuel element plant for the manufacturing of low enriched fuel elements for research reactors and the delivery of about 30 elements to the DR3 reactor. Finally, in an area that epitomizes technical advance based on earlier basic research, a new powder metallurgy process has been developed for the manufacturing of high strength, light metal matrix composites with good high-temperature stability.

Symposia and Workshops

An important activity in the Department during 1989 has been the organization of the »Tenth Risø International Symposium on Metallurgy and Materials Science«; the subject was 'Materials Architecture. The Scientific Basis for Engineering Materials'. The 4-day symposium at Risø was followed by a one-day symposium in Copenhagen on the theme »Materials in Modern Society«. The 4-day scientific symposium was attended by about one hundred scientists from universities, industry and from government laboratories. On the last day of the symposium many participants from within Denmark were also present, representing the Parliament, the Central Administration and the Media. The titles of the Risø symposia in the period 1980 - 1989 are given in Table 1. The subject for 1990 has been chosen to be »Structural Ceramics - Processing, Microstructure and Properties«; this symposium is cosponsored by the Danish Centre of Engineering Ceramics. For the benefit of Danish materials scientists and industry, the Department held a one-day meeting on the subject of advanced materials and metals technology. The number of participants was about 140. Another activity was the organization of a workshop in Silkeborg, Jutland on the subject of »Radiation Damage Correlation for Fusion Conditions«. This 5-day workshop was arranged in collaboration with PSI, Switzerland and was attended by about 50 scientists.

Internal stress measurement by neutron diffraction of thick-walled tube. CrMo steel component which had been exposed to an internal pressure of 2500 bar.



Education

Education and training are an important responsibility of the Department, and many undergraduates and graduates from the Danish Technical University and from abroad have received academic and practical training in the Department. Several post graduates have also studied in the Department. For details see Chapter 6. At present, educational activities are being increased and extended to include the training of technical employees. Part of this activity has been carried out under the auspices of the COMETT Programme (EEC) in collaboration with Brunel University of West London. A new venture has been the planning and teaching of a comprehensive course for technicians and engineers on materials properties, processing and product design. This course is under the sponsorship of the Danish Ministry of Education and has been planned together with a number of research institutes, universities and technical schools in Denmark.

Visits

The Department was host to a number of guest scientists, whose names are given in Chapter 9. In addition the Department received around four hundred visitors representing many different groups and technical societies in Denmark.

Teaching of materials testing techniques was part of the COMETT course. This EEC programme was carried out in collaboration with Brunel University of West London.

Publications and Reports

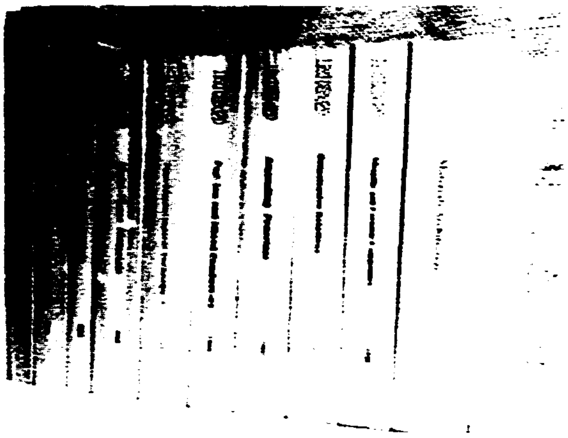
The results of the scientific and technical work are primarily reported in international scientific journals and conference proceedings. However, a relatively big part of the contract work appears in reports of limited circulation and in confidential reports. A list of publications and lectures is given in Chapter 10 - 12. The abstracts of these publications are published in a special report, »Metallurgy Department, Publications 1989«. In order to ensure a broad circulation of the results of the research, a number of overview papers have been written in Danish and circulated widely to interested parties.

Risø International Symposia on Metallurgy and Materials Science 1980 - 1989

Year	Title
1980	Recrystallization and Grain Growth of Multi-Phase and Particle Containing Materials.
1981	Deformation of Polycrystals: Mechanisms and Microstructures.
1982	Fatigue and Creep of Composite Materials.
1983	Deformation of Multi-Phase and Particle Containing Materials.
1984	Microstructural Characterization of Materials by Non- Microscopical Techniques.
1985	Transport-Structure Relations in Fast Ion and Mixed Conductors.
1986	Annealing Processes - Recovery, Recrystallization and Grain Growth.
1987	Constitutive Relations and their Physical Basis.
1988	Mechanical and Physical Behaviour of Metallic and Ceramic Composites.
1989	Materials Architecture. The Scientific Basis for Engineering Materials.

Concluding Remarks

During 1989 the Department was able to achieve a rather firm scientific, technological, and economic basis on which to build its activities in the coming years. Important elements of this basis are the extensive participation in the Danish Materials Technology Programme, the strong involvement in the national programme on fuel cells and the participation in many programmes under the auspices of EEC. The national and international interest in the work of the Department, both within the areas of fundamental and applied research, provide good promise for a future where national research increasingly will be affiliated with joint European programmes.



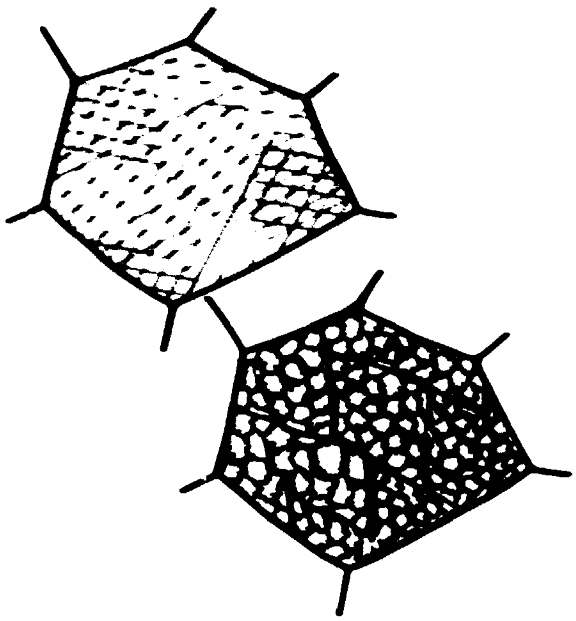
The Department collaborates extensively with industry, research organizations and universities.

2. MATERIALS SCIENCE - Modelling and Characterization

Efforts to improve the inherent properties of materials are based on our ability to characterize, understand and finally to modify microstructure. The research in this area, although of a fundamental nature, is often initiated in response to specific technological and engineering demands for new and improved materials. The research themes in Materials Science are therefore closely related to the applied programmes within the Department. Much of this basic research is carried out in close cooperation with colleagues from universities and government research laboratoaries around the world.

Current research activities are concentrated in the following areas:

- Quantitative modelling of deformation microstructures texture and recrystallization.
- Mechanical properties and modelling related to the behaviour of single crystals and polycrystalline metals as well as metal matrix composites, dispersion strengthened materials and polymer matrix composites.
- Defects and microstructure caused by irradiation with energetic particles. Mechanical properties of materials for fusion reactors.
- Ion conductivity in solid oxide electrolytes for use in fuel cells.
- Development of advanced techniques for the characterization of materials. Neutron diffraction and small angle neutron scattering, electron microscopy and positron annihilation.



Dislocation structure and slip line pattern in grains where individual regions deform by less than five independent slip systems. Dense dislocation walls (DDW's) delineate volumes containing ordinary equiaxed cells.

2.1. Polycrystals Deformation

The evolution of the cold deformation microstructure has been studied for medium to high stacking fault energy, single phase materials. (This research is carried out in collaboration with The Danish Academy of Engineering, University of Virginia, and Sandia National Laboratory, California). Microscopic strain accommodation for polycrystalline materials has been considered and it is suggested that grains subdivide during deformation on a smaller and smaller scale and that each volume element is characterized by an individual combination of slip systems. This deformation model involves that, within each volume, fewer slip systems operate than specified by the Taylor criterion for strain accommodation; however, collectively the slip in a group of adjacent volumes acts so as to approximately fulfil this criterion. This slip model leads to a reduction in the flow stress as the number of intersecting jogs is reduced. Thus, at a given strain, the stress required for further deformation is reduced. Crystal deformation based on a reduced number of slip systems will therefore be energetically favourable. The number of slip systems should not, however, be too low since strain accommodation will be increasingly difficult as the number of slip systems is reduced. Furthermore, a certain number of slip systems must be operative in order to ensure the build up of cell walls as low energy dislocation structures in which near neighbour dislocations mutually screen their individual stress fields. Although the number of slip systems arising from these competing effects is difficult to predict, it must be expected that the number of slip systems operating will be substantially less than that required by the Taylor model.

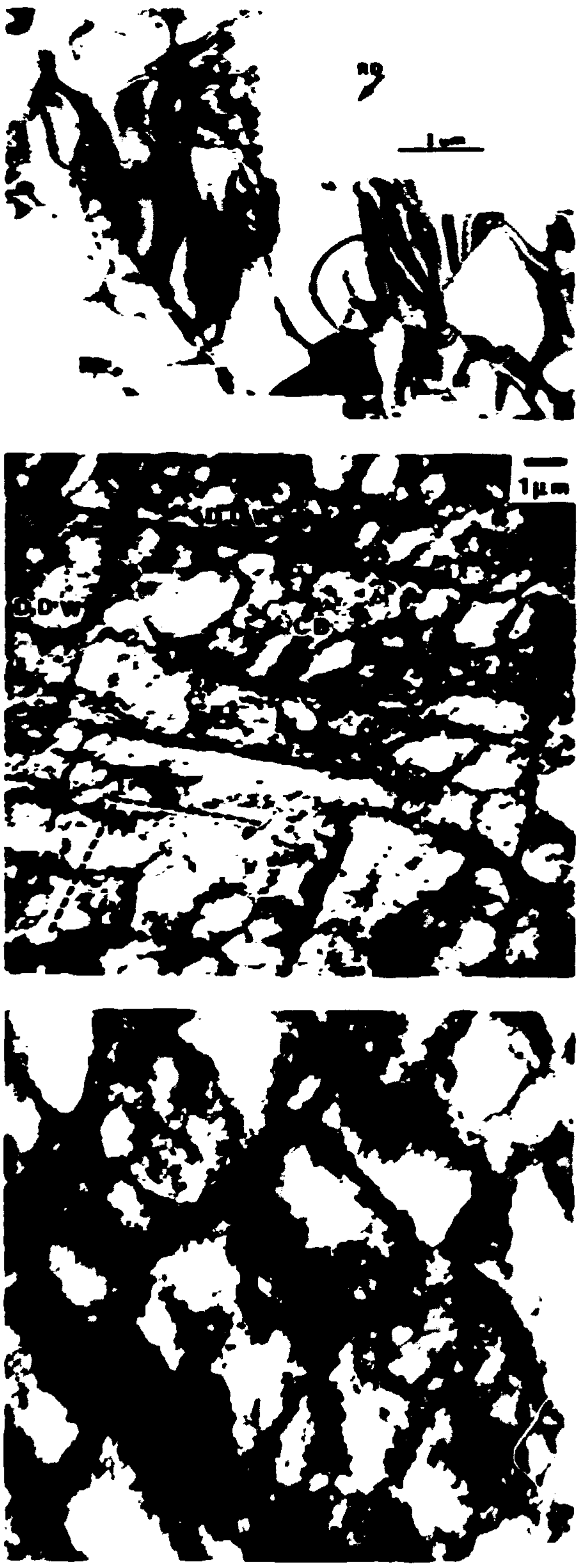
Based on the principles mentioned above a number of microstructural observations (especially of aluminum, nickel, and copper) have been analysed and the various dislocation arrangements characterized, especially various types of microbands (see Figure). This detailed structural examination and characterization have led to the conclusion that the deformation pattern and the microstructural evolution are comparable in medium and high stacking fault energy materials and that the general principles for polycrystal deformation mentioned above, to a large extent, explain the experimental observations.

2.2. Quantitative Modelling of Microstructure and Texture

Quantitative modelling of the development of deformation texture was first made at Bell Laboratories and Risø National Laboratory (independently) more than 20 years ago, so it is by now a rather mature discipline. Over the years a number of laboratories throughout the world have devoted a very substantial effort to develop the models, and they have now reached a high level of mathematical sophististication. However, the models are, with very few exceptions, based on very simple assumptions about the microstructure. This sets a limit to the accuracy in texture predictions that the models can provide. But there are more serious implications than that: the single-minded dedication to texture modelling has meant that there are practically no quantitative models available for the equally important evolution in microstructure. The ideal situation would be simultaneous modelling of the microstructural and the textural development, and the Metallurgy Department is actively engaged in an attempt to approach this situation.

This attempt finds inspiration in a number of recent microstructural observations which clearly demonstrate that the microstructural evolution is not as simple as assumed in the texture models: »organized structures« like dense dislocation walls, microbands and bundles of twin-matrix composite form im the grains of deformed materials. Examples, taken from work in which the Metallurgy Department is engaged, are shown in the figure opposite. Recent investigations show that the mechanical anisotropy of deformed materials is partly caused by the microstructure - and not solely by the texture as previously assumed. This obviously adds to the importance of modelling of the microstructure.

Transmission electron micrographs of deformation structures in aluminium, nickel and copper. Dislocation arrangements and microbands were examined in detail.

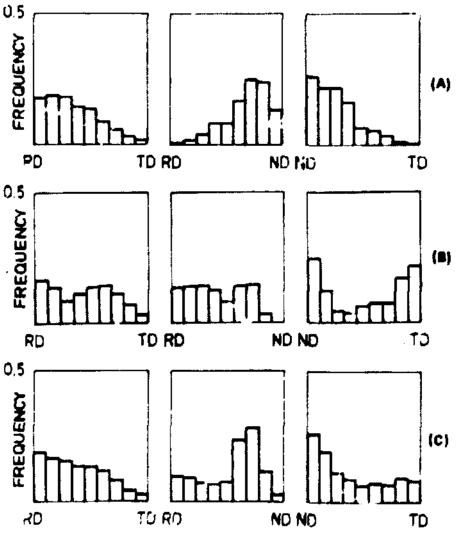


In the field of recrystalliztion the situation is different. Some models focus on microstructure (grain morphology) and neglect texture. Others focus on texture neglecting microstructure. At the Metallurgy Department work on the inclusion of texture in microstructural modelling is now in progress.

Models for Deformation

In face-centred cubic metals (or rather alloys) with low stacking fault energy, »bundles« of twin-matrix composite are the predominant microstructural features. Because of the simple crystallography of the twinning process it is relatively easy to include the formation of twin lamellae and hence the bundle formation in deformation models which at the same time produce simulated textures. The figure below shows the simulated orientation distributions of twin lamellae for the Taylor model and the modified Sachs model together with the experimental orientation distribution. The orientation distributions are given as the distributuions of twin lamellae projected on the rolling plane, the longitudinal plane and the transverse plane. The figure shows that the modified Sachs model provides an adequate simulation, where the Taylor model does not. The simultaneous texture simulations reflect the same pattern: the texture simulated with the modified Sachs model is much closer to the experimental texture than that simulated with the Taylor model. Other microstructural simulations (not to be described in detail) show that the grains with twin-matrix bundles (about 50% of the grains) deform by predominant single glide, which means that strain continuity is maintained by accommodation in the other grains - within the framework of the modified Sachs model. Thus, for brass it is, by now, possible to do simultaneous modelling of the microstructural and texture development and to get consistent results.

Computer-simulated orientation distributions of twin lamelae for the Taylor model (B) and Sachs model (C) compared to the experimentally measured orientation distribution (A) in brass, rolled 40%.



Grains in deformed aluminium subdivide in regions (»cell blocks«) with different slip systems operating. As a first approximation it appears that there are two alternating families of cell blocks so that a cell block belonging to one family borders two cell blocks belonging to the other family. Various quantitative models have been formulated for the description of this situation. In one model the grains follow the macroscopic strain as required in the Taylor model, but the cell blocks do not. This may be achieved either with 4 active slip systems in each family of cell blocks or with 5 in one family and 3 in the other. In principle the model may also be used for texture simulation. In practice there is a rather great number of solutions (a high degree of ambiguity), and therefore no texture simulations have been made so far. Thus, modelling within the framework of a more realistic microstructure is possible for aluminium but actual simultaneous modelling or simulation of the microstructural and the textural development requires additional structural information.

A Model for Recrystallization

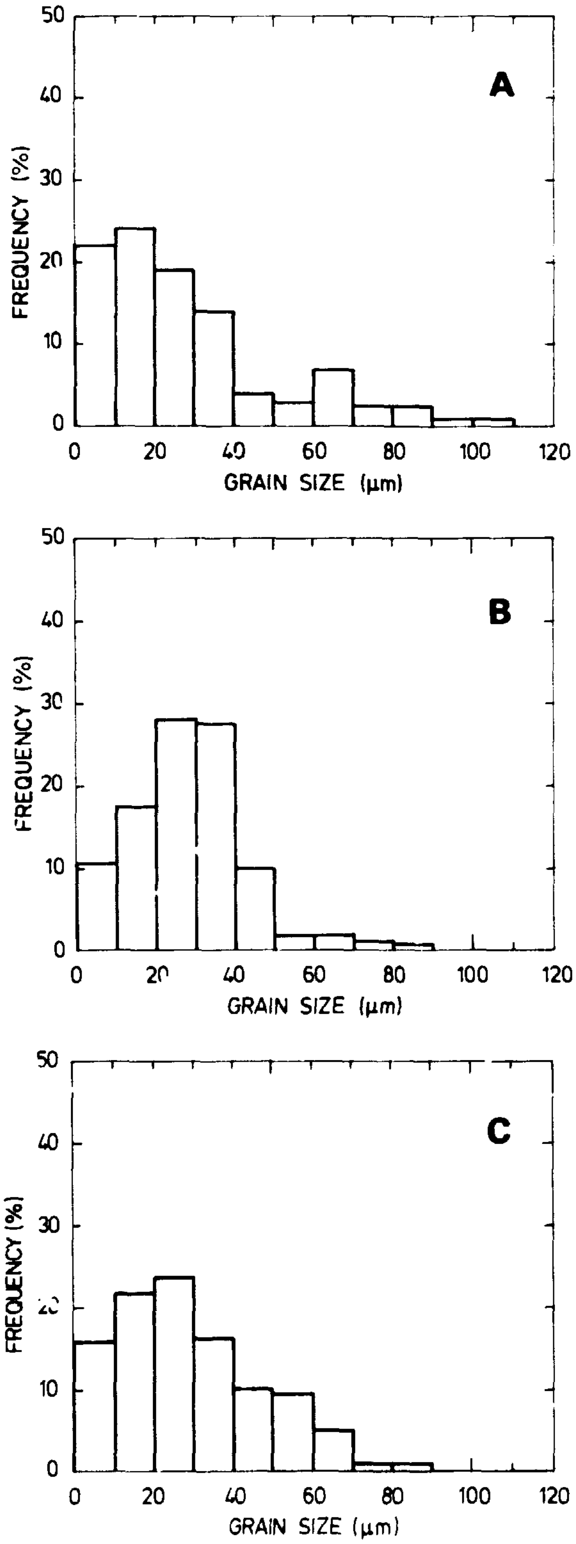
The model considers the formation and subsequent growth of up to 5 types of nuclei, representing 5 texture components. The nuclei may form with arbitrary distributions in space, and time, and each of the five types grows with a specific growth rate. The growth rate may vary in time (after nucleation) and it may be different in the different directions. Growth ceases where two grains impinge but continues in all other directions. The model thus keeps track of the grain morphology of the recrystallizing material and of the crystal orientation in each of the grains, i.e. the model can do simultaneous simulation of the microstructural and the textural development.

The model has been used to simulate grain morphology, recrystallization kinetics and texture in commercially pure aluminium rolled to 90% reduction and recrystallized at 253°C.

The recrystallization texture as measured can be described by three components: cube, retained rolling and random. The input parameters for the model were obtained from microstructural investigations. Nucleation could, for all three texture components, be described as random in space and concentrated at time zero (site saturation). Two approximations were used for the growth rates (which was the same in all directions): constant rate or a rate which decreased linearly with time. The microstructural investigations indicated that the growth rates decrease, but the rate of decrease is rather ill-defined. Both the number of nuclei and the growth rate were different for the different texture components. The grain morphology and the recrystallization kinetics derived from the model agree best with the experimental observations when the growth rates are taken to decrease with time. The observed and the simulated grain size distributions after complete recrystallization are shown in the figure opposite. The texture derived from the model agrees best with the experimental texture when the growth rates are taken to be constant. The reason for this decrepancy seems to be that the observations do not provide sufficiently accurate growth rates.

The critical dependence of the experimental input parameters illustrates the nature of the present models for recrystallization. The overall process, viz. the nucleation of new grains and their subsequent growth into the deformed material, is beyond discussion. But the models are based on purely empirical descriptions of the nucleation and growth processes. It is obvious that a very substantial effort is needed to provide a physical description of these processes. It is equally obvious, on the other hand, that already models at the present stage, as that described here, represent a great step forward compared to the earlier analytical models. They are particularly useful as tools for the study of the nucleation and growth processes, whereas their use as tools for prediction of microstructures and textures is at present more questionable.

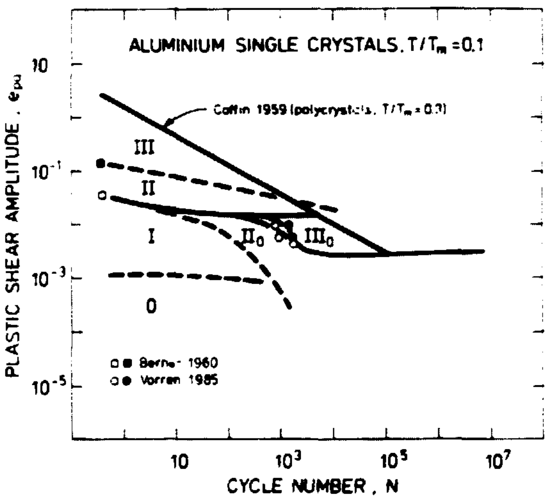
Grain size distributions for fully recrystallized aluminium · measured and theoretical; measured (A), simulat. with constant growth rate (B) and simulated with decreasing growth rate (C).

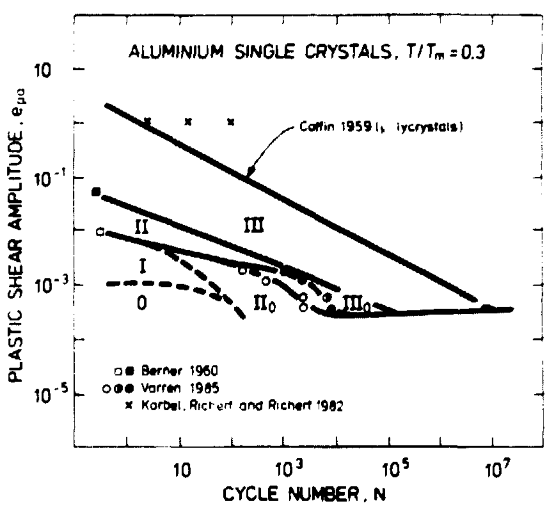


2.3. Modelling and Mapping of Cyclic Plasticity

Cyclic plasticity controls the nucleation and slow growth of the fatigue cracks, which limit the life of otherwise flawless metallic components under oscillating mechanical loads. It would therefore be of considerable importance in mechanical design to have a realistic and comprehensive set of constitutive equations for cyclic plasticity. Here we consider cyclic plasticity at »low« temperatures, that is, temperatures below about half the absolute melting temperature. In general, a first step in the development of constitutive equations for low-temperature plasticity is to identify genuine hardening stages, that is, stages within which the governing physical mechanisms remain the same. The fatigue mechanism maps for aluminium single crystals represent such genuine hardening stages for cyclic plasticity as two-dimensional patterns of fields in diagrams of (constant) plastic strain amplitude versus cycle number. The maps are »model inspired«; but it is fair to say that they are as yet largely empirical, since the physical mechanisms in the individual fields are incompletely understood. The mechanisms of tensile work hardening in field II, for example, remain controversial. Nevertheless, central aspects of the hardening processes appear to be well understood, at least in simple model systems like copper with undeformable tungsten fibres. Consequently, the maps were constructed within the theoretical framework of the so called »composite models«, in which dislocation-dense regions are represented as hard ellipsoidal inclusions in a softer matrix. The framework also covers the effect of polycrystallinity.

Fatigue mechanism maps for aluminium single crystals together with field patterns in the aluminium map.





Model inspired mapping offers numerous useful experimental checks on the resulting constitutive equations. The reason is that in order to be completely successful a constitutive equation must not only be valid within its domain of application. It must also be capable of predicting the position and size of its domain of application. It was therefore encouraging that a composite model supplemented with a cross-slip model could be shown to predict that an increment of temperature ΔT displaces the top boundary of field III_0 to lower amplitude by an amount which is simply proportional to ΔT. It turned out that the field patterns in the aluminium map behave in accordance with this theoretical prediction. A comprehensive investigation, involving cyclic experiments on Cu at 77 K and an exhaustive literature survey, revealed that the prediction is confirmed by cyclic experiments on a range of other face-centred cubic alloys. A similar prediction of the top boundary of field II by the theory is also experimentally confirmed. Furthermore, related models suggest that the position of the fatigue life curve terminating field III_0 depends upon cross-slip among other mechanisms. It therefore appears that a combination of semi-empirical mapping, composite models and cross-slip models may lead to a comprehensive set of dis-ocation-based constitutive equations, which can account realistically for work hardening and fatigue in metals and alloys. As they stand, the maps do not show a field IV, corresponding to the large strain behaviour in monotonic deformation. However, further studies using more complex deformation modes, like cyclic torsion, may allow the evolution of the microstructure and texture during cyclic straining in field IV to be explored and plotted onto the maps.

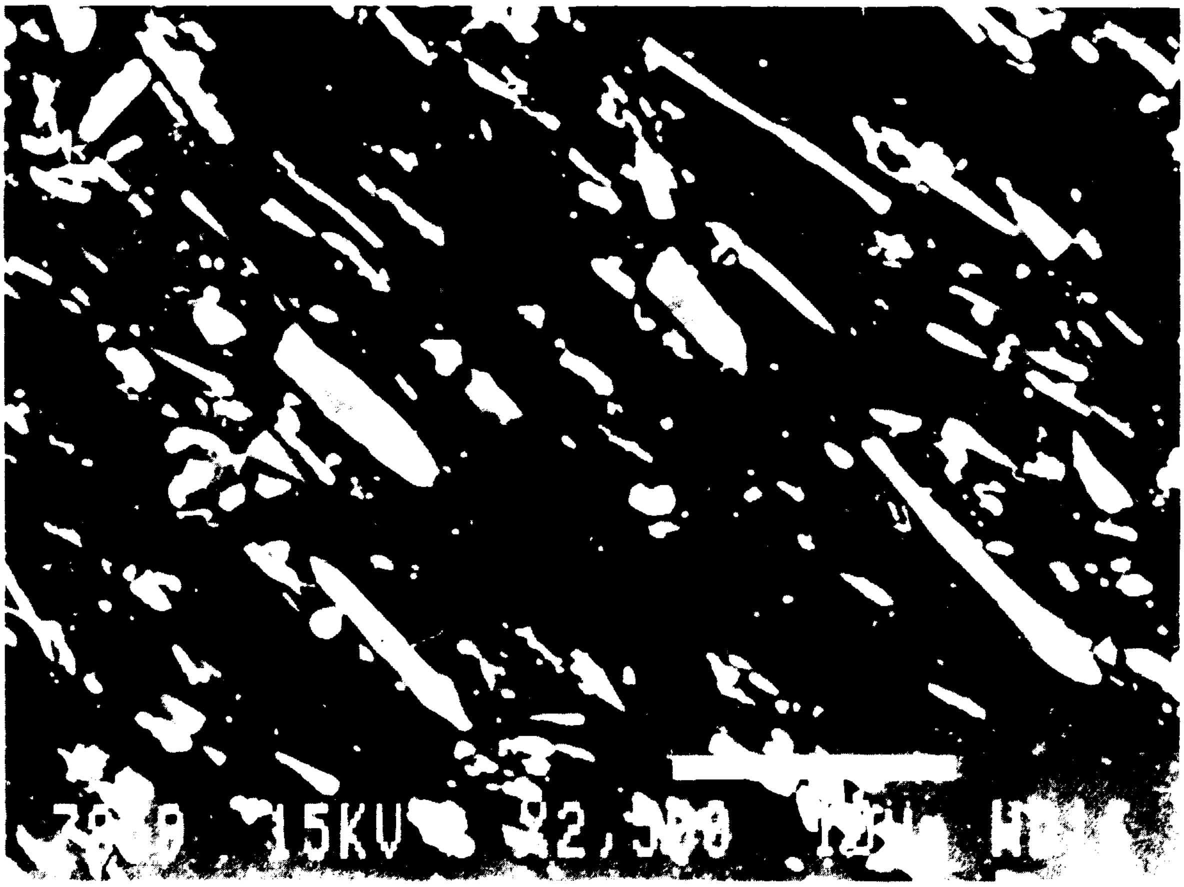
2.4. Metal Matrix Composites

Recrystallization of Al/SiC

The study has been concentrated on the recrystallization behaviour of cold-rolled silicon carbide whisker reinforced aluminium composites also containing fine aluminium oxide particles. The cold-rolled structure and the changes introduced by recovery and recrystallization have been examined. The effect of metallurgical parameters such as the volume concentration of silicon carbide whiskers and fine aluminium oxide particles has been investigated.

The presence of the whiskers enhances microstructural development during cold deformation. Deformation zones form locally around the whiskers and such zones can be strong nucleation sites. The fine alumina particles retard nucleation and inhibit dynamic recovery. The number density of nuclei and the recrystallization temperature are considerably affected by the parameters of fine alumina particles and degrees of deformation. Homogeneous recrystallized grains are obtained when particle stimulating nucleation takes place and nuclei grow by high angle grain boundary migration. The grain size is reduced with increasing whisker concentration, decreasing alumina content, increasing deformation and annealing temperature. For composites containing a large content of whiskers and small content of alumina particles, the recrystallized structure consists of recrystallized grains and recovered subgrains.

Scanning electron micrograph of Aluminium metal matrix composite strengthened with short fibres of silicum carbide and small particles of aluminium oxide.



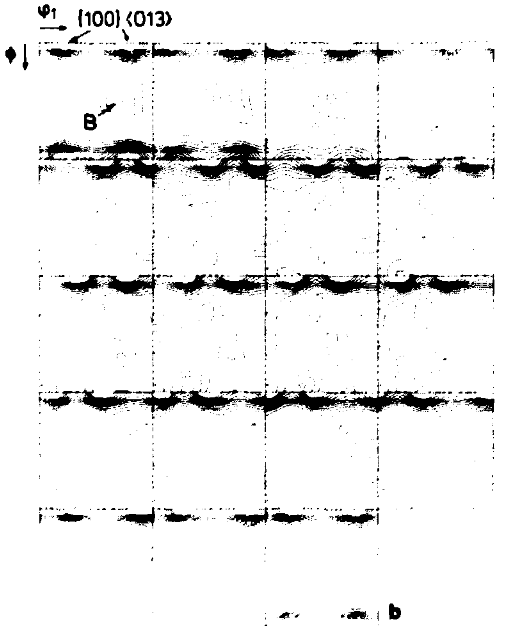
Texture of Al/SiC

The textural development during cold rolling and subsequent annealing has been studied by neutron diffraction for Al reinforced with 2 vol% SiC whiskers. When combined with microstructural investigations such data give important information about the deformation and recrystallization processes taking place in MMC.

The cold rolled texture of Al-SiC is qualitatively rather similar to that of other medium to high stacking fault energy fcc metals. However, compared to a similar material without SiC fibres, the peak intensities are significantly weaker. Such a weakening or smearing out of the deformation texture has previously often been observed in materials containing large (>0.1 μm) second phase particles, and has been ascribed to the development of deformation zones with large local lattice rotations surrounding the particles.

The recrystallization texture is characterized by a very strong rotated cube component ({100} <013>) and other components fairly randomly distributed in the orientation space. This is not one of the typical recrystallization textures in fcc metals and alloys. To understand the reasons for the development of this new texture, microtexture and electron back scattering techniques have been used.

Very strong rotated cube texture in recrystallized Al/SiC.



These techniques give simultaneous information about microstructure and crystallographic orientation and thereby more information about the nucleation and growth processes is obtained. It was found that the nuclei of {100}<013> orientation relate to the {100}<011> texture present in the starting and in the deformed material, and that they originate from clusters of fibres with large lattice rotations within the deformation zone. In the fully recrystallized state the average size of the {100}<013> grains is larger than that of the other grains, which correlate well with the observed strength of the {100}}<013> recrystallization texture. The reason for the more limited growth of grains with other orientations is at present not fully understood and further investigations are underway.

Internal Stresses in Al/SiC

In metal matrix composites (MMC) such as aluminium based materials reinforced with ceramic fibres or particulates (e.g. SiC), residual stresses can have great influence on the mechanical behaviour of the composite. Thermal induced residual stresses are built up during production of the materials due to a mismatch in coefficients of thermal expansion; for example, the mismatch between aluminium and SiC corresponds to a factor of 6.

Residual stresses in multi phase materials such as the Al/SiC group of materials, can be studied using a neutron diffraction technique providing information on the bulk residual strains in the different constituent phases. Such experiments can be performed under controlled tension/compression of uniaxial test specimens, and strains can be measured within ~ 5 - 10 min and with a strain resolution generally better than 1 x 10^6.

A first generation tensile machine for in-situ neutron diffraction strain measurements has been implemented, and a second generation machine, prepared for thermal loading as well, is under construction.

One example of studies of internal stresses in MMC composites are measurements on aluminium specimens reinforced with 5 and 10 Vol.% SiC whiskers of aspect ratio ~ 5. Whisker alignment was determined through texture studies discussed previously (2.3b) and the lattice strain response of both phases under controlled tensile loads was studied using the neutron diffraction technique.

The observed lattice strain response for the aluminium matrix and the SiC whiskers as well as the macroscopic strain response measured by strain gauges. The neutron diffraction technique monitors elastic strains whereas the strain gauges measure total strains, hence it is seen that the aluminium matrix ceases to build up elastic strains beyond 0.1% strain whereas elastic strains reaching 0.4% are observed in the SiC whiskers. This study, performed in close collaboration with the Material Science Department of Cambridge University, illustrates how the neutron diffraction technique can be applied to monitor the difference in mechanical behaviour of different phases in a composite material.

Much departmental effort is focused on MMC materials both on modelling of mechanical behaviour, on optimisation of production and manufacturing techniques as well as on creep phenomena. Within all these areas, valuable information on the composite behaviour can be gained from neutron diffraction strain measurements, and coming studies will include both thermal and mechanical cycling as well as measurements during creep. Such experiments are expected to provide an enhanced understanding of the behaviour of composite materials.

Mechanical Behaviour of MMC

The mechanical properties of Al/SiC_w composites have been examined by tensile testing at room temperature. The microstructure in the undeformed state and after straining to 1- 10% has been analyzed by TEM. As compared to the Al material without SiC, the composites show an enhanced work-hardening rate during straining, which is related to a greater dislocation density and a faster build up of dislocation substructures. An analysis of the various strengthening contributions from grains and subgrains, dislocations generated during cooling and work hardening, dispersion strengthening and long range internal stresses is important in order to understand the strengthening mechanisms in these materials.

The effect of material and P/M processing parameters on the structure of composites such as the aspect ratio, alignment and distribution of the SiC whiskers and the microstructure of the matrix has been studied . Some correlation between structural parameters and mechanical properties has been estabished. An optimization of the P/M process with respect to the mechanical properties has been proposed.

2.5. Deformation of Polymer Matrix Composites

Reinforcement of polymers with strong and stiff fibres is a very efficient method for improvement of the mechanical performance of polymers. The properties of these polymer matrix composites under mechanical, thermal and chemical (corrosive) loadings are studied by several techniques. The aim is to establish knowledge of the behaviour of polymeric composites as a basis for the use of the materials in design. The special direction of the work is towards long term performance under demanding conditions.

Creep Behaviour

The studies involve creep properties of polymeric composites of glass fibre/polyester and carbon fibre/epoxy. Both experiments and modelling are carried out with the microstructural parameters being volume fraction of fibres, fibre length/diameter ratio and fibre orientation. The models have been established and are valid for all fibrous composite materials; the models account for fibre volume fraction, length/diameter ratio; and will be further developed to include fibre orientation.

Experiments on pure epoxy are performed at 90°C; the creep curves of strain versus time show a visco-elastic region with decreasing creep rate. An analytical description for this is being developed. The creep curves show a visco-elastic region with decreasing creep rate. An analytical description for this is being developed. The creep curves show a very brittle behaviour of the epoxy with no final creep region (stage III).

Experiments on glass/polyester laminates with fibre orientations ± 10° are made at 150°C with stresses of 45 - 50 MPa. The creep curves show practically no visco-elastic region (stage I), and a rather long region of constant creep rate (stage II). The analysis of creep rates versus stress does not follow the simple model.

Fatigue Behaviour

The studies involve a detailed investigation of glass/polyester laminates with various fibre orientations: 0°, ±10°, ±45°, ±60°, and 0°/±45°. This family of materials are considered model-materials for practical wing blades for wind turbines.

The experiments are made at room temperature and at frequencies of 5-10 Hz. The maximum (initial) strain in the fatigue loading is plotted versus the logarithm of number of cycles to failure. A possible fatigue limit may exist at cycles of 10^7 or beyond; the importance of such a fatigue limit is that loading below such a limit will mean (essentially) infinite lifetime, and thus constitute a (very) safe design criterion.

Fracture of glass fibre reinforced composite test specimen after fatigue testing.

During fatigue loading the stiffness, typically the bending modulus (E-modulus), will decrease. The moderate reduction in modulus at medium lifetimes may be used to define a design limit based on an allowable reduction in modulus during operation of wing blades (on a wind turbine).

The mechanisms leading to reduction in modulus are based on the formation of cracks in the material; models are being developed to analyse such mechanisms.

Tensile Deformation

The studies involve tensile testing at various temperatures to characterise the polymers and the polymeric composites. The pure epoxy is brittle at temperatures below the glass transition temperature and will often show premature failure due to (existing) minute cracks. At higher temperatures (90 - 120°C) the behaviour is more ductile leading to a maximum in load during tensile testing.

Models for the stress-strain curves are being developed, to describe the pure epoxy and allow calculations to be made for the corresponding composites. The models will also allow extrapolation for the (low temperature) stress-strain curves beyound the point of (premature, brittle) failure.

Deformation of Thick Laminates

The classical theory for the elastic deformation of laminates is based on the assumption of a laminate thickness which is small relative to other dimensions. Polymeric composites are increasingly used in components and products where this thickness is not small. Work has been made for thick laminates, including finite element calculations with higher order displacement functions. The element has a 5th order polynomial distribution of the transverse displacement and a 3rd order variation of the in-plane displacements; the model has excellent convergence properties. The model has been verified against other numerical solutions on simple geometries, and limits for the validation of thin plate theory have been established by an evaluation of the strain energy from the different deformation modes (bending, in-plane deformations, and transverse shear).

2.6. Irradiation Defects - Fusion Materials

Irradiation of metals by energetic particles removes atoms from their lattice sites, leaving vacancies and creating interstitial atoms. Very energetic particles can, in addition, induce nuclear reactions and generate gaseous and non-gaseous impurity atoms, changing the composition of the irradiated metal or alloy. The created defects may diffuse together to produce nanometer-size defect clusters; their presence changes mechanical, thermal, magnetic and electrical properties. The *fundamental study of such defect clusters and of* the generated microstructural changes lies at the heart of physical metallurgy; the diffusion processes and their consequences are closely related to temperature effects and property changes not involving irradiation. The work in this area comprises both theoretical and experimental efforts, the applied experimental techniques being transmission electron microscopy, positron annihilation, and possibly small angle neutron scattering.

These techniques are being developed to give fundamental information close to the atomic level. This information is necessary to the other areas of our work in which mechanical properties of materials for future fusion reactors are being studied.

Gold irradiated with 600 MeV protons at PSI, Switzerland. The smallest visible 'residual cascade defects' are less than 1 nm in diameter. Weak beam transmission electron micrograph.

Correlation of Fast Neutron, Spallation Neutron and Fusion Neutron Irradiation Damage Structures in Copper and Gold

(In Collaboration with Paul Scherrer Institute, Switzerland)

Irradiation of pure metals at ambient temperatures by fast neutrons, spallation neutrons, fusion neutrons and 600 MeV protons allows cascade damage structures to be studied which result from recoils of 10^4-10^6 eV respectively. High resolution weak beam transmission electron microscopy has been used to document the densities and size distributions of the defects produced by these irradiations in pure copper and gold. The results are being analysed in order to be able to correlate microstructural changes with those to be expected in future fusion power reactor components.

Cascade Damage and Cavity Nucleation

(In collaboration with KfA Jülich, F.R.G. and Harwell Laboratory, England)

The study of the possible role of cascade production in the nucleation of cavities has been continued. The cavity density and its temperature dependence are calculated in terms of diatomic nucleation assumed to occur homogeneously as well as in cascade induced vacancy aggregates (CIVAs). For helium diffusion between CIVAs, two very different mechanisms are considered, namely the fast self-interstitial/He replacement mechanism and the slow vacancy mechanism for substitutional He.

The slow vacancy and the fast self-interstitial/He replacement mechanism for helium diffusion yield substantially different temperature dependencies of the effective helium diffusivity as well as cavity density. The present work confirms the conclusion reached earlier that the presence of cascades would contribute to the cavity density only if helium atoms were to diffuse very fast between defect clusters via the replacement mechanism. For such fast helium diffusivities, cascades would result in a very strong temperature dependence of the cavity density below 0.5 T_m (T_m = melting temperature). However, available experimental data on fcc metals and alloys do not show such a temperature dependence. Hence, it is concluded that at least in fcc metals and alloys cascades do not affect helium diffusion and cavity nucleation in any significant way.

The Concept of Production Bias and its Role in Void Swelling

(In collaboration with Atomic Energy of Canada Ltd., Canada)

In the rate theory treatment of void swelling both vacancies and interstitials are assumed to be produced continuously in time and uniformly in space. Under cascade damage conditions, however, both vacancies and interstitials are produced in a highly localized and segregated fashion. Consequently, spontaneous clustering of both kinds of defects is likely to occur in the cascade zone.

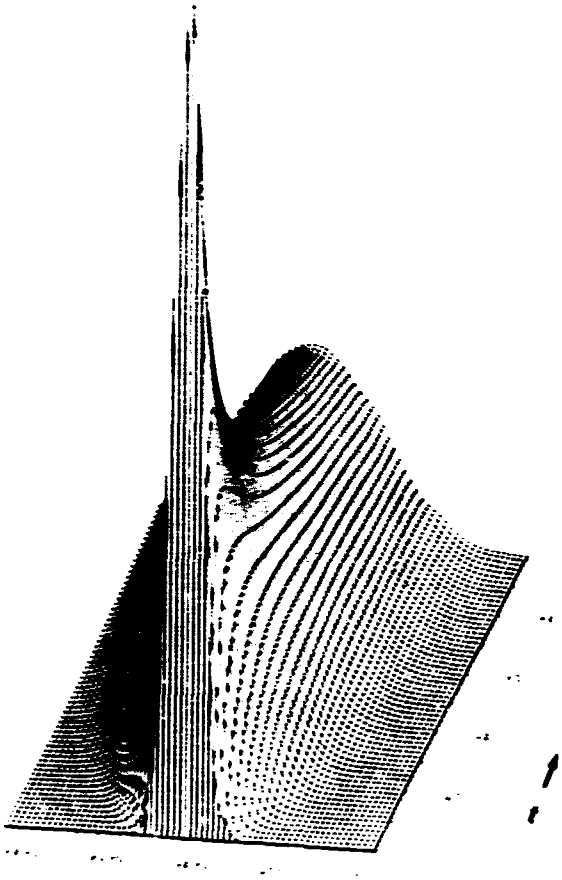
A model has been formulated which takes into account the effects of both vacancy and interstitial clustering on defect accumulation rate (e.g. void swelling rate) under cascade damage conditions. The consideration of interstitial clustering in the cascade yields a production bias which is found to be a potent driving force for void swelling in addition to the one provided by the dislocation bias. The bias mechanism giving rise to production bias is very different from that involved in the concept of dislocation bias.

Since the production bias is recoil energy dependent, a detailed knowledge of the interstitial clustering as a function of recoil energy is required in order to be able to predict the spectrum effects on void swelling behaviour.

Recombination and Clustering of Interstitials Under Cascade Damage Conditions

(In Collaboration with Atomic Energy of Canada Ltd., Canada and Battelle Pacific Northwest Laboratory, U.S.A.)

A programme has been initiated to develop a diffusion-based methodology to study the space- and time-dependent recombination and clustering of point defects generated during the cascade process.



Evolution of mono-interstitial distributions in a cascade. Plotted on the z-axis is the number of single interstitials in a spherical shell, radius R (jump distance) and time t (number of jumps).

The reason for adopting this diffusion-based continuum approach is that the global microstructural evolution is too large a problem to be treated in terms of interactions of individual atoms.

As a first step, an attempt has been made to perform analytical and numerical calculations of the space and time dependent diffusion recombination and clustering of interstitials. Preliminary results show that it is computationally feasible to determine the recombination and clustering of interstitials, at least for a single cascade. Two interesting results from the present calculation are that a significant portion of the interstitials may be immobilized in the cascade zone by clustering, and that of the interstitials escaping the cascade zone, a significant portion may be di-interstitials which may have different diffusion properties from the mono-interstitials.

Displacement Damage and Helium Effects in Copper and Copper Alloys

Low oxygen grade copper and copper alloys are being considered as candidate materials for divertor components in fusion devices such as NET (Next European Torus) and ITER (International Thermonuclear Experimental Reactor). The materials used in the divertor components will be exposed to an intense flux of fusion (14 MeV) neutrons. The resulting displacement damage and helium generation may cause void swelling and helium embrittlement.

A programme has been initiated to study these effects in copper and copper alloys. Materials to be used in this programme are: Cu (OFHC), Cu-Al_2O_3, Cu-Cr-Zr and Cu-Ni-Be. Different types of specimens of these materials are being irradiated with fast neutrons (at Risø) and 600 MeV protons (at PSI, Switzerland) at the NET/ITER relevant temperatures. Pre- and post-irradiation mechanical properties (tensile and low cycle fatigue) and electrical conductivity of these materials will be determined. Pre- and post-irradiation microstructures will be characterized using transmission electron microscopy, small angle neutron scattering and positron annihilation techniques.

Specimens in the form of 3 mm discs are being irradiated in FFTF (Fast Flux Test Facility) reactor at Hanford (U.S.A.) to dose levels of up to 10 dpa. Some of the specimens irradiated in FFTF to a dose level of ~30 dpa have already been examined.

In order to evaluate the effects of helium generation rate at a given temperature, a series of irradiation experiments are scheduled to be carried out at KFK Karlsruhe. Two of such experiments have already been carried out and the resulting microstructures are being evaluated.

2.7. Solid Electrolytes - Ion Conductivity

Oxygen ion conductors, mainly zirconia and ceria based systems were investigated with respect to ionic conductivity as a function of temperature, composition and presence of impurity phases in the grain boundaries. X-ray and SEM characterisations were also carried out. A puzzling behaviour of Mg-stabilised zirconia, some with SrO additions, and Fe_2O_3 and CuO added YSZ was observed: Although quite different the in nature of dopants, impurities and phase distribution one could observe that all samples »obeyed« a common law of compensation correlating pre exponential term and activation energy for ionic conduction.

The Department participated in a world wide round robin test on »Ionic Conductivity in Fluorite Type Oxides« applying impedance spectroscopy. A new frequency response analyser (10^{-5} Hz to 32 MHz) for measurement of bulk, interfacial, and electrode impedances was purchased and implemented.

2.8. Characterization of Microstructure

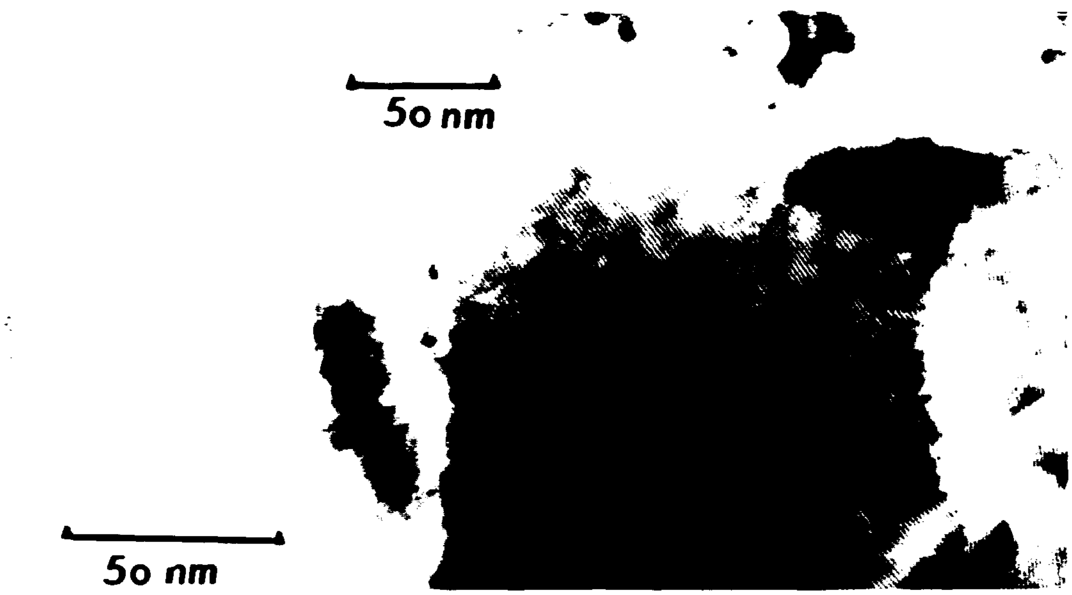
Electron Microscopy

The relation between structure and properties plays an important role in many of the Department's projects. Two transmission electron microscopes - JEOL 2000FX and JEOL 100C - and one scanning electron microscope - JSM 840 - are available for structural analysis. All microscopes are equipped with energy- dispersive X-ray spectrometers for chemical analysis; the JEOL 2000FX is furthermore equipped with an electron energy loss spectrometer.

Software has been developed for on-line microtextural studies with the electron backscattering pattern attachment on the scanning electron microscope and is now used routinely.

Microstructural work has been performed for a number of the Department's own projects. This work is reported under the individual projects. During the year microstructural analysis has also been done for a number of industrial customers. This work has included e.g. examination of corroded thermocouples and corroded tubes and chemical analysis of high temperature superconductors. Electron microscopical assistance has also been given to the Physics and the Information Technology Departments.

In collaboration with the Physics Department, studies have been made of the orientation and morphology of Pb islands on Ge (111) and Si (111) surfaces. The growth is of the Stranski-Krastanov type (2D layer + 3D clusters). An example of a transmission electron micrograph of a Pb island on Ge is shown in the figure below.



Transmission electron micrograph showing epitaxy of Pb islands on (III)Ge using the Moiré fringe technique. Note the low-angle grain boundary dislocations.

Neutron Scattering

Neutron scattering gives statistical information which can supplement microscopy investigations. The penetration depth of neutrons in most metals and alloys and in many ceramic materials is several centimeters. Thus, the neutrons give information about bulk properties or can be used as an NDT technique to sample volume elements inside larger components. At present three experimental techniques are used for materials science investigations: texture, internal stress and small angle neutron scattering.

The texture equipment allows the measurement of one pole figure in about 10 minutes and the development in single texture components can be studied in-situ with the time resolution of the order of seconds. As a new development, the equipment is now controlled by a VAX computer which eases the data evaluation. Examples of investigations carried out during the year are a) effect of strain and particle parameters on the deformation and recrystallization textures in metal matrix composites, b) comparison between deformation/recrystallization texture in pure Cu deformed by channel die compression and by cold rolling, c) textures in rapid solidified Al materials and d) effect of grain size on texture development in tensile deformed Cu.

The implementation of the internal stress equipment with a linear position sensitive detector and a sample robot (X-Y-Z translation plus two mutually perpendicular rotations) has been completed. The strain profile (type I internal strain) through a structural component can be determined with a spatial resolution of ~1 mm, a strain resolution of $\sim 1 \times 10^{-4}$ and in typical steel samples the penetration depth is 2 - 3 cm. Measurements of internal strain profiles near weldments and near a crack tip in a CTS specimen have been carried out. Further, measurements of internal strains in thick walled tubes exposed to large internal pressure have been compared to finite element calculations. Information about the performance of the applied constitutive model is thereby obtained. The average strain for complete samples (type II internal strains) has been studied during thermal and mechanical loading of metal matrix composites. For this type of measurement, the strain resolution is also $\sim 1 \times 10^{-4}$. A new version of the equipment with four motorized beam definers for measurements of strain in very thin layers (<1/2 mm) of, for example, solid oxide fuel cells is at present being designed.

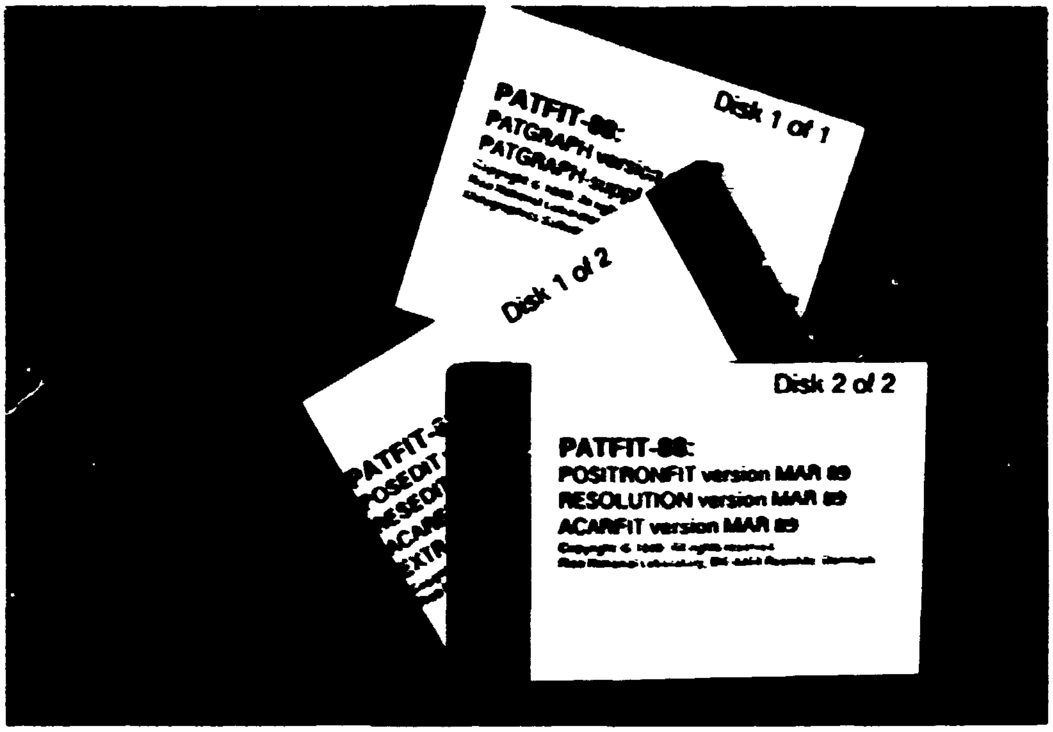
The small angle neutron scattering (SANS) facility has been upgraded and a new 12 m long, very versatile instrument is now in operation. The two dimensional detector can be moved continuously from one to six meters from the sample and the sample to source point distance can be varied from one to six metres by inserting neutron guides in the collimation section. This allows structure elements (such as voids, precipitates etc.) on the scale 10-1000Å to be studied with the optimum flux for a given resolution. In the materials science area the SANS has been used for studies of krypton bubbles in copper. The size distribution and the volume concentration of Kr bubbles were determined for a series of samples annealed at various temperatures. Further, the SANS equipment was used for determination of the surface area of cavities in a series of high temperature fatigued copper samples. This was to gain information about the growth law for the cavities.

Positron Annihilation

In studies of defect-properties of metals it is useful to apply different experimental techniques which may provide complementary information. One of the defect sensitive techniques is the so-called positron annihilation technique (PAT) in which the sample under investigation is bombarded with positrons (antiparticles to electrons). Their annihilation with electrons in the sample results in the emission of gamma-quanta. The time and angular correlation of these gammas can be measured and give experimental information about electron density and momentum in the sample in the vicinity of the positrons. Since positrons are attracted to vacancy type defects (e.g. single vacancies, vacancy clusters, vacancy-impurity clusters etc.) the presence and characteristics of such defects can be detected by PAT, because at one such type of defect the electron density and momentum normally are different from those of the bulk metal and of other defects.

Measurements have been initiated in order to study the development of helium bubbles during annealing of He-implanted iron. In this collaboration with Jülich, F.R.G., the PAT results shall be compared with measurements by Small Angle Neutron Scattering (SANS) on the same samples. Iron foils of 0.1 mm thickness were He-irradiated (at ISPRA) to 2000 appm He, uniformly distributed through the sample and positron measurements carried out during isochronal annealing up to 520°C where bubble coarsening begins to take place. A clear difference is observed in the defect configuration between the »beam-entry side« of the foil and the »back« side. On the former side, the positron lifetimes are longer and with higher intensities than on the back, showing a defect density as well as vacancy to He ratio being higher near the front than near the back. The He density in the bubbles near the back side is estimated to 13×10^{28} m^{-3} for the as-prepared samples, dropping to $11 \times 10^{28} m^{-3}$ after the 520°C anneal. Rough estimates for the bubble radii and concentrations are 28Å, 2×10^{-7} and 50Å, 4×10^{-8}, respectively.

Fast measurement of texture by the neutron scattering technique; the pole figure.



Last year a program package was developed for the analysis of PAT data. Until now this package has been acquired by more than 35 laboratories in more than 21 countries.

For comparison with SANS measurements on Krypton bubbles in copper (see other section) a number of PAT lifetime measurements were carried out on identical samples to the ones used for SANS. The results were found in detailed agreement with previously published data.

In order to study vacancy interaction with alloying elements in zirconium, two sets of annealing experiments have been carried out on zircalloy-2 and zirconium - 2.5 wt% niobium electron irradiated to a dose of about 2 x $10^{17}e/cm^2$ (1.3 MeV) at liquid nitrogen temperature (collaboration with Chalk River Nuclear Laboratories, Canada). In the zircalloy-2 which contains 1.15 wt% Sn, vacancies produced at low temperatures apparently disappear below 220K, probably due to recombination with migrating interstitials. (In the Zr-Nb alloy no such effect is observed, probably because of interstitial-Nb binding). Up to about 350K another defect configuration (maybe vacancy impurity complexes) is dominant, while above 350K some clustering of vacancies (or complexes) takes place. In the Zr-Nb alloy, defect complexes disappear between approximately 225 and 350K while vacancies recover or form complexes with impurities above approximately 350K.

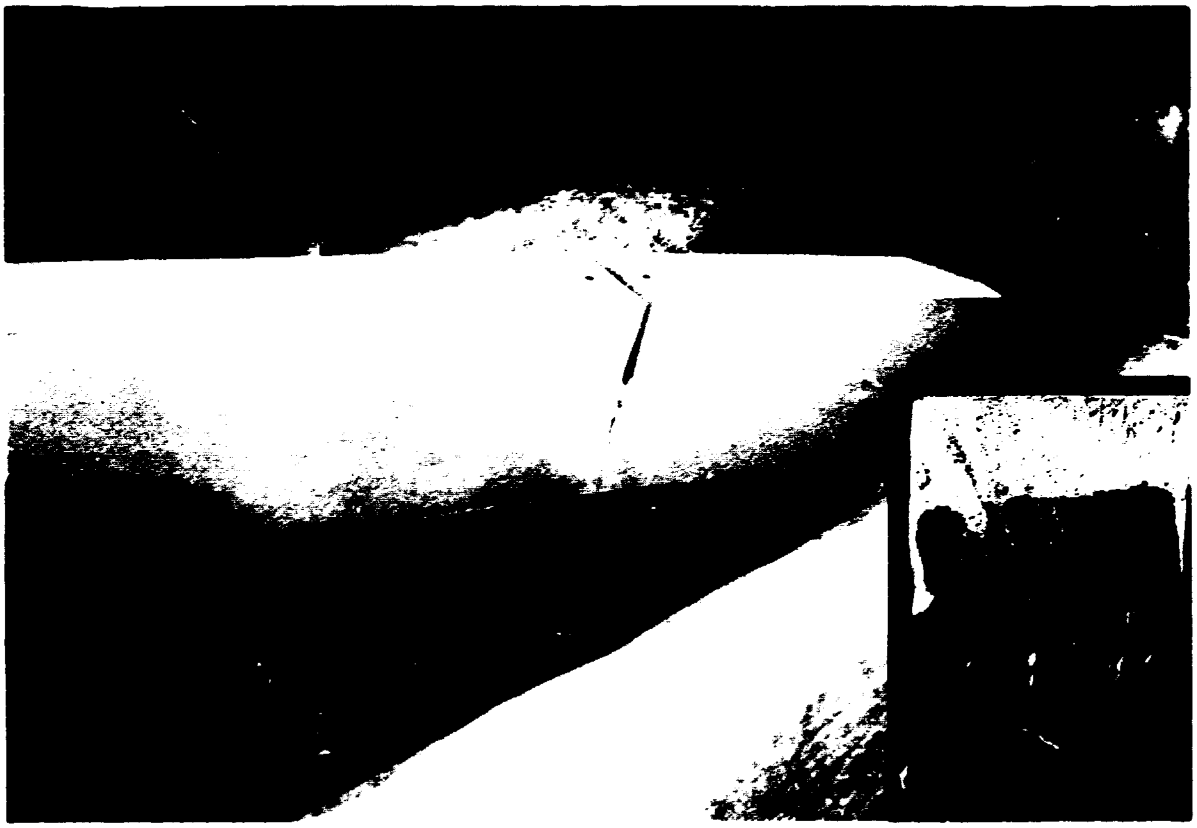
3. MATERIALS ENGINEERING - Design and Testing

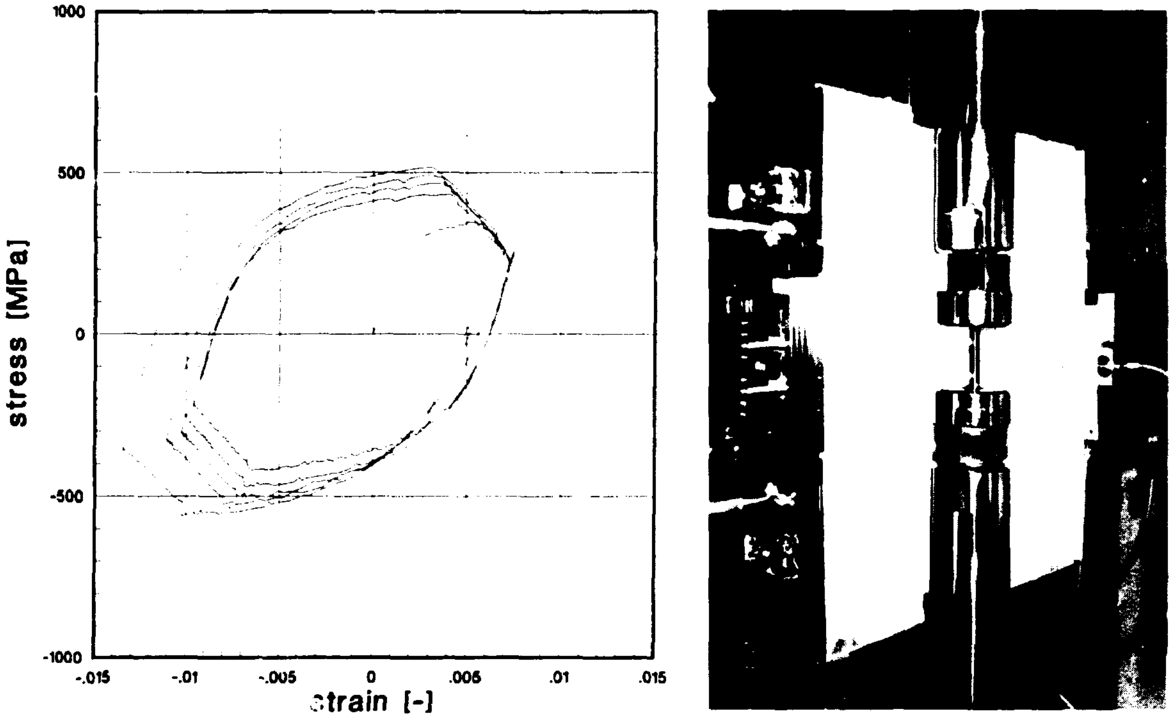
A thorough knowledge of the mechanical properties of engineering materials is essential for the design of advanced components and structures. Of special interest are materials for the oil and gas sectors and composite materials for windmills, helicopters and lightweight pressure vessels as well as engineering ceramics. The research activities in Materials Engineering are centred around structural mechanics analysis of destructive and non-destructive materials testing procedures. A considerable number of the projects are carried out in close collaboration with Danish and European industrial partners.

Current research activities are concentrated on the following areas:

- Improvement of component design and service life prediction based on static, dynamic and fatigue testing results. Materials studied are steels, metal matrix and polymer matrix composites and engineering ceramics.
- Environmental effects due to in-service surface degradation by aggresive fluids or vapours, humidity and high temperatures.
- Detection and analysis of processing flaws in engineering ceramics and composite materials.
- Detection and analysis of cracks in metals and ceramics and delamination in polymer composites.
- Development of computer programmes for the aquisition, analysis and management of data.

Testing of fatigue crack growth in large steel specimens. The insert shows the fracture surface after end of test fracture in liquid nitrogen.





Axial stress versus axial strain in thin-walled aluminium tube; square loading cycle. The furnace, above right, with associated spring-suspended extensometer and quartz rods, allows uniaxial testing at high temperatures.

3.1. Structural Mechanics

The structural mechanics work in the Metallurgy Department applies computational mechanics to predict the behaviour of structures and components and the material behaviour of these. Furthermore, it is utilised in the analysis of results from materials testing in order to interpret the test results and ultimately to transfer them to design values.

Thermo-Mechanical Behaviour of Steel

A 3-year research program on the plastic thermomechanical behaviour and modelling of steel has been concluded in 1989. The program was funded by the Danish Agency for Technology and performed in collaboration with MAN-B&W Diesel and The Technical University of Lund. Two different types of steel have been studied experimentally at temperatures ranging from room temperature up to 400°C aiming at obtaining data to be used in the mathematical models of constitutive behaviour. The materials were a CrMo steel and a stainless steel; cyclic tests were performed in both tension and compression for different temperatures, and biaxial tests at room temperature. Two theoretical models have been developed parallel to the experimental work:

The two surface model, which is based on a formulation, where the bounding surface exhibits combined kinematic and isotropic hardening. This model is formulated with a particular view to obtain predictions of complex cyclic loading. The model, developed in collaboration with the Technical University of Lund, will be implemented in the commercial finite element code SOLVIA at Risø.

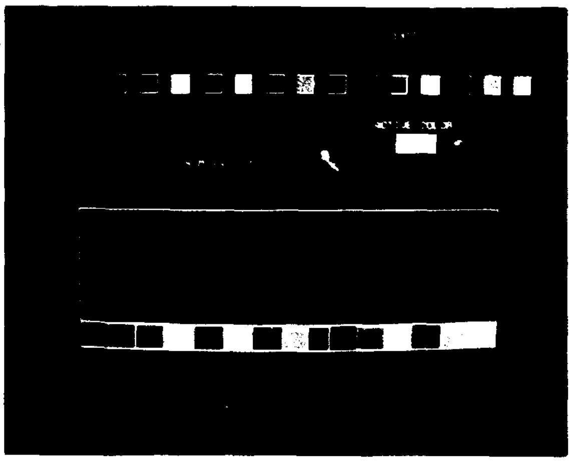
The shear-plane model, in which it is supposed that a finite number of shearing planes exist which pass through a given material point in different directions. Each plane is treated independently and combined by means of numerical integration. This model has been developed by MAN B&W Diesel and implemented in reduced form in their finite element code HIFINEL.

The experimental part of the project comprised testing of 82 uniaxial specimens at different temperatures and load cycles, and 20 biaxial specimens at different loading patterns at room temperature.

Computational mechanics are applied for the analysis of specimens used in materials testing and for design analysis of components and structures. Composite materials test results may not be indicative of the actual performance of the material as used in a structure, this being especially pronounced for the polymer based composite materials. The methods are also being applied to fibre reinforced ceramic materials.

Thin cylindrical composite tubes for testing a specific manufacturing technique have been analysed with the purpose of designing hydraulic testing equipment and to define the valid evaluation zones for subsequent sectioning of the specimens. Load introduction problems involved in the testing of fibre reinforced ceramics are another example of the use of the analysis as support for materials testing.

Delamination is an important defect type in polymer based laminates, and the capability of the finite element method to model contact problems has been initiated in order to model the stiffness reduction due to this type of defect. Such stiffness reduction may be important in certain structural applications of composite materials.



Accurate and fast ultrasonic scanning of materials has been improved. As an example of a more user friendly interface for data evaluation, the figure shows how we, by means of a mouse, directly can choose the colours for different echoheight (or distances) from an histogramic representation of all data from a scan.

3.2. Non-Destructive Ultrasonic Testing of Advanced Material

Several new projects on non-destructive characterization of advanced materials have been started both in connection with two of the Danish materials centres (Centre of Engineering Ceramics, Centre of Polymer Composites) and in a European (BRITE) project on fibre reinforced materials for helicopters.

Accurate and fast ultrasonic scanning of materials has been improved. As an example of a more user friendly interface for data evaluation, the figure opposite shows how we, by means of a mouse, directly can choose the colours for different echoheight (or distances) from an histogramic representation of all data from a scan.

Both the scanning system and the ultrasonic equipment have been updated. The technical reason is to be able to inspect at higher frequencies (50 MHz or higher) and thereby get better resolution for inspection of advanced materials from the national and international projects. Having examined and tested different equipment, we found the best fulfilment of our needs in the HFUS 2000 instrument, which now has been bought. Resolution of the single layers in a carbon-epoxy plate made of 8 prepregs is improved using a 50 MHz transducer compared to a 25 MHz transducer and also the old S80 ultrasonic instrument. A small stepper-motor driven scanning system has been included in the set-up which allows smaller samples to be studied and more precise scanning than the 1/10 mm resolution achievable in the hydraulic scanning system.

The system is built around a 386 based personal computer. The measurements can be performed by one of several ultrasonic instruments. For the moment we can use the HFUS for both measurement of echo-amplitude and distance between echoes. A precision thickness instrument (Panametrics) can also be used for accurate dimensional measurement. The file-structure of the data files are optimised to the actual measurement. Scanning movement can be performed on a hydraulic scanning system or a steppermotor-system. The speed and the size of the samples to be inspected as well data structure and inspection criteria, differ between the two systems.

3.3. Management of Huge Amounts of Data in Nuclear Fuel Projects

Evaluation of nuclear fuel experiments involves large and comprehensive amounts of information. In the Third Risø Fission Gas Project (see for instance Risø-R-547, pp. 39 - 41) such information included fabrication data and drawings from fuel vendors, irradiation data from commercial power reactors, data and drawings from refabrication at Risø, in core measurements from irradiation of refabricated, instrumented fuel pins in the DR3 reactor and data from a variety of post-irradiation measurements carried out in the Risø Hot Cells.

It is important to have access to a full, constantly updated data set for both evaluation purposes and planning of new experiments and initiation of measurements. Anticipating the volume of data within the Third Risø Fission Gas Project an electronic data bank was established at the start of the project in 1985. Upon reception of data from abroad and completion of measurements at Risø, all information was placed on a central computer, accessed by staff members of the project group from their PC's via a standard low speed network. An uncomplicated organization of the data was chosen to enable sharing of the information with project staff having less experience with computers. The information (text, numbers of reference to data files containing large data sets) was placed in ASCII tables for plain reading off the computer screen or printing; data files referenced in the tables were formatted for direct import in standard spreadsheets, drawings were stored in formats allowing import in standard CAD programmes. The responsibility for updating information and maintaining traceability of data was distributed between staff members of the project group.

The system has presently been in operation for five years. Even with the limited amount of time invested in the construction and management of the data bank, a good overview of project status and a high efficiency of data evaluation has been achieved. This is in spite of the volume of data which approaches 500 Mbytes. A detailed description of the data management system is given in the publication »Management of Huge Amounts of PIE Data« (see paragraph 10).

3.4. Engineering Ceramics

Materials research within the centre project on processing science and technology was concentrated on the development of new ceramic materials with the aim of improving fracture toughness. Very promising results were obtained with the ceria stabilized zirconias (Ce-TZP) either pure or mixed with Al_2O_3. For the composite Ce-TZP/20 wt% Al_2O_3 prepared by mixing coprecipitated Ce-TZP (containing 12 mole% CeO_2) with a commercial Al_2O_3 powder, powders exhibiting both excellent pressing and sintering behaviour could be obtained. Fixed samples of this composition showed fracture toughness values of 28 $MPa^{1/2}$ but still with a flexural strength (determined by the 4-point bending technique) of 650 - 675 MPa. Similar strength values could be obtained by mixing yttria partially stabilized zirconia (TZ3Y) with Al_2O_3 fibres, but in this case no improvement was observed in the fracture toughness compared to that of pure TZ3Y. Finally, a Ph.D. project has been started on fibre reinforced ceramics. Initially, the thermomechnical behaviour of glass-matrix reinforced with continuous nicalon (SiC) fibres will be studied and the properties of this system will be modelled.

In the centre project on the testing of ceramic components for specific applications, the Department is particularly involved in testing of ceramic seals, especially for water pumps. The wear properties are of prime importance for this application, and for the characterization of abrasive wear of ceramic materials the so-called microwear techique has been developed. In this subproject, characterization of the abrasive wear of different types of ceramic materials has been started.

An important application of especially zirconia ceramics is as solid electrolytes in oxygen sensors. Used for in-situ measurements in industrial furnaces these are, however, very often exposed both to severely corrosive atmospheres as well as to severe thermal shocks. In a sub-project within this testing programme, oxygen sensors produced from either commercial tubes or from tubes produced in the processing project are being tested to establish the properties which are limiting industrial environments at high temperatures for these sensors. The department is also involved in development of joining methods by brazing and diffusion bonding and in the devleopment of NDT by ultrasonic techniques. Both of these activities are described separately.

Abrasive wear testing of engineering ceramics. The micro-wear technique is currently being used to determine wear resistance of ceramic valves and bearings.

An important property of ceramic materials is the fracture toughness (K_{IC} and in the material research of these materials it is very important to have reliable techniques for the measurement of this property. Fracture toughness values are usually determined by the Vickers inductative technique, but as this technique is generally not precise, a technique based on controlled slow crack growth has been developed.

Sensor Ceramics

Research was continued on development and testing of oxygen sensors. Besides long-term laboratory testing, corrosion testing of sensor materials in an industrial furnace for production of porcelain was also carried out. Furthermore, SO_2-sensors based on NaSiCon-electrolytes have been developed and tested. Provided both electrodes are painted with a sodium sulphate solution before the measurements, reliable measurements can be obtained with this sensor. Finally, SnO_2 sensors for measurements of CO also was developed.

4. MATERIALS TECHNOLOGY
Fabrication and Processing

The manufacture of advanced materials components often requires new processing, fabrication and joining techniques. Pilot plant studies of the production of fibre reinforced polymer composites, fine-powder metallurgical components and thin ceramic layered structures demand the construction of specialized equipment. This research and development also provides test specimens of new advanced materials for other programmes of the Department. The research activities in Materials Technology involve the manufacture of components of polymer matrix composites, engineering ceramics, prototype solid oxide fuel cells and fine- powder metals. Brazing and bonding techniques are being applied to a variety of these materials. The research programmes are carried out partly within the three Danish centres, Advanced Technical Ceramics, Powder Metallurgy and Polymer Composites, and partly in collaboration with other Danish and European research organizations and industrial partners.

Current research activities are concentrated in the following areas:

- Manufacture of advanced polymer composites by a variety of filament winding and fibre pre-form methods.
- The fabrication and joining of thin ceramic layers and oxide electrodes for solid oxide fuel cells.
- Inert gas atomization to produce fine metallic powders of new aluminium and iron-based alloys.
- Advanced technical ceramics processing.
- Brazing and joining techniques for powder metallurgical steels, aluminium alloys, superalloys, ceramics and ceramics to metals.

4.1. Manufacturing Processes for Advanced Composite Materials and Products

The involvement of the Metallurgy Department in manufacturing of continuous fibre reinforced plastics serves mainly three purposes: a) study of the fundamental principles of filament winding, autoclave processing, and resin transfer moulding, b) fabrication of test specimens, and c) development of prototype components.

The processing equipment consists of a computer-controlled filament winding machine, a hot-air high-temperature high- pressure autoclave, and equipment for resin transfer moulding.

A fundamental study of the filament winding technique has been completed. The influence of the process parameters (winding speed, fibre tension, temperature, and thickness of laminate) on the quality of wound laminates of fibre reinforced thermoset was examined. Three different winding processes were investigated: wet-filament winding where a welling drum is immersed in a bath of matrix material; wet-filament winding where the matrix material is supplied in a quantity continuously controlled by the amount of fibre wound onto the mandrel; and dry-winding followed by a resin transfer technique. Filament winding of high- temperature thermoplastic composites inside a heated oven was also a part of this study. The study was a confidential project and therefore further details cannot be stated here.

An ultra light racing canoe for use in the Marathon races has been designed and manufactured in collaboration with students from the Danish Engineering Academy. The over-all length and width of the canoe are respectively 5.2 m and 0.75 m, and the weight of the canoe is only 6.3 kg, which is only 40% of the weight of a standard canoe of mahogany and 63% of the weight of a commercial canoe built in carbon fibre composites. The canoe is built in an advanced fibre composite sandwich material. The skin layer consists of a hybrid carbon/aramid woven cloth in an epoxy matrix, and the sandwich core material is PVC foam. The carbon fibres give the canoe the required stiffness and the aramid fibres give the required resistance against impact.

Ultra light racing canoe for use in Marathon races. The construction is based on a fibre composite sandwich of cross-woven carbon and aramid fibres around a core of foamed PVC. Design and construction was carried out in collaboration with students from the Danish Engineering Academy.

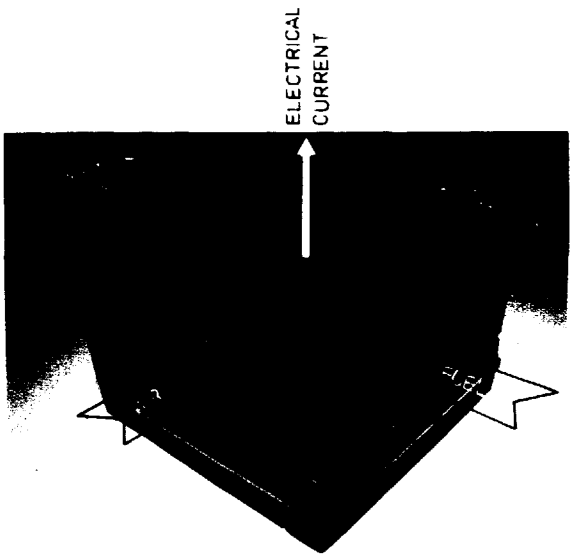
Two new projects on manufacturing technology for thermoplastic composites with continuous fibres have started late in 1989. The first project is on filament winding and the second one is on fibre pre-forms. (Fibre pre-forms are a pre-shaped fibre structure which has not yet been consolidated to the final structural component. Fibre pre-forms can be woven, knitted, braided or stretched together). The purpose of the projects is to examine the possibilities, limitations, and the characteristics of a fabrication technique where the material (fibre + matrix) is either wound onto a mandrel at room tepmerature or placed in a mould at room temperature in the form of a fibre pre-form, and consolidated by a subsequent autoclave process.

Another purpose of the filament winding project is to develop a computer aided design program (for use on a PC-computer) for filament wound structures. In the first step of this work the program will be limited to consider only geodesic path on objects which are symmetric rotationally.

4.2 Fabrication of Solid Oxide Fuel Cells

Solid oxide fuel cells (SOFC) are predicted to generate electricity and heat at 900 - 1000°C with a higher efficiency for electricity production than in conventional power generation. The fuel may be hydrogen, hydrogen-carbon monoxide mixtures and hopefully the cells will also operate directly on natural gas. Solid electrolyte materials as well as air- and fuel electrode materials are therefore being studied.

Techniques for fabrication and joining of these materials in the form of thin ceramic layers are under development. The methods encompass tape casting, RF-sputtering, slurry and gel-techniques. Candidate materials are studied both in thin and bulk form, the latter being produced by combinations of cold pressing, isostatic pressing and machining of green ceramic bodies.



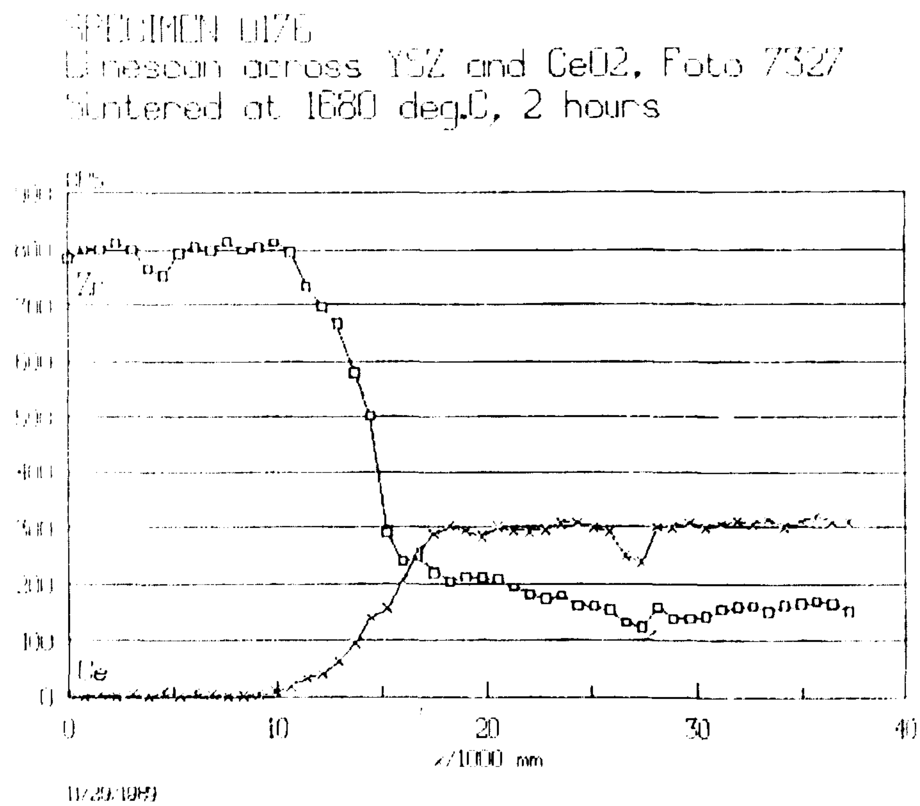
Model of a Solid Oxide Fuel Cell for efficient production of electricity.

Thin (0.1 mm) yttrium stabilised zirconia ceramic (YSZ: the solid electrolyte in the fuel cells) is being tape cast routinely. A comparative study of the ionic conductivity of this Danish material with different tapes from British, American and Japanese competing SOFC projects proved that the Danish make performs very well. Tape casting of air electrode material of the lanthanum- strontium-manganite type is done in collaboration with a Danish industry.

Electrode studies have shown oxide-based electrodes to be much more suitable than metal-based electrodes for the direct oxidation of methane. The strategy has thus been to modify such binary and ternary oxide formulations in order to obtain both good electrical and electrocatalytic performance. More basic studies of these mixed conducting oxides (oxygen ion - and electronic conductors) were also undertaken.

Ceria is a potential anode material. Therefore the interface betweem yttrium stabilized zirconia and ceria sintered together at various temperatures and sintering times was studied by SEM and TEM. The concentration profile across the interface in the sintered samples was measured by energy-dispersive X-ray analysis. A Kirkendall effect was observed with cavities developing close to the position of the original interface. The phases developed in the interdiffusion zone were examined by transmission electron microscopy.

Energy Dispersive X-ray Analysis technique applied in a scanning electron microscope study of ceramics used in solid oxide fuel cells.



4.3. Advanced Technical Ceramics

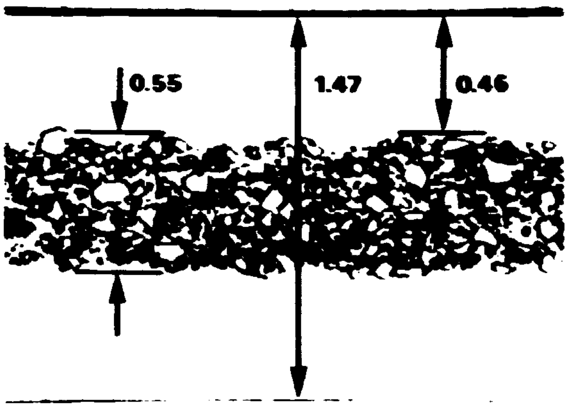
In the Danish materials technology development, advanced technical ceramics have been recognized as an important class of materials of direct interest to the Danish industry. Within this programme, the »Centre of Advanced Technical Ceramics« was therefore created in Spring 1989. This centre, which is managed by one of the staff members of the Department, involves a close collaboration between six major Danish industries and three research institutions including this Department. The yearly budget for this centre is DDK 10 million. In the centre, research and development in the ceramics field are carried out within the following projects in which the Department is also participating: a) Processing science and technology for oxide- based ceramics; b) Testing of engineering ceramics for specific applications; c) Development of Piezo-electric ceramics; d) Information and education. Besides these projects the Department is also engaged in projects on sensor ceramics, i.e. ceramic materials combining good mechanical properties with a reasonable ionic conductivity.

4.4. Powder Metallurgical Processing

Manufacturing of Low Enriched Uranium Fuel Elements

For many years fuel elements for the Danish DR3 reactor have been fabricated at Risø. These elements contained high-enriched uranium (HEU) with more than 90% U_{235}. During recent years our fabrication facility has been rebuilt to enable production of a new type of element containing low-enriched uranium (LEU) with less than 20% U_{235}. The uranium cores for these LEU elements are made powder metallurgically from a mixture of U_3Si_2 and Al-powder.

In 1989 reconstruction of the production plant was finished and fabrication of elements was started on a production scale. During the year more than 500 compacted fuel cores were produced and a total of 32 elements were delivered to DR3.



Micrograph of UO_2 fuel encapsulated in aluminium sheet for DR-3 fuel elements.

Inert Gas Atomizer

As part of a project under the recently started Danish Materials Technological Programme (see »Introduction«) the Metallurgy Department has worked on the construction of an inert gas atomizer for the production of fine metallic powder. The fundamental design of the equipment is based on the atomizer at Massachusetts Institute of Technology, Boston, U.S.A., but a number of significant improvements have been incorporated. The project is being carried out in collaboration with the Danish Technological Institute and Niro Atomizer A/S.

The equipment at Risø is anticipated to be in service by the end of 1990. The melting chamber can be pressurized in order to decrease the relative variations in pressure, which are often found in the metal flow tube outlet. The melting range of the atomizer furnace will allow light weight metals like aluminium alloys as well as alloys based on iron to be manufactured.

The size range of the powders to be produced by the atomizer is anticipated to start around 1 μm, with a significant yield at as low as 20 μm. These particle sizes will ensure rapid solidification properties of the powders and even in some cases production of metallic glass particles.

The Risø inert gas atomizer is prepared for later extension with facilities for spray-compacting (sometimes called Liquid Dynamic Compaction) and it is also prepared for extended in situ determination of particle sizes and velocities utilizing laser measuring techniques. The latter may well become of great interest in conjunction with spray compacting.

Ultrafine Powders

Ultrafine powders, i.e. powders of particle sizes in the nanometer range, do not yet have any industrial relevance. One reason for this is that the production rate is counted in grams per day. Nevertheless, this kind of powder has unique properties, that may well lead to practical applications. One such property could be the very strong sintering activity, which may be utilized for, say, joining of PM-parts solely by sintering.

In collaboration with the Laboratory for Technical Physics at the Technical University of Denmark, the Metallurgy Department has studied the behaviour of ultrafine powders. The powders were produced at the Technical University in various compositions of iron and nickel. Because of their extremely fine sizes they react vigorously with oxygen. Therefore the reduction properties in hydrogen were studied at Risø. The results obtained until now indicate that the oxide on the ultrafine powders is removed both more easily and at lower temperatures, than is found when using coarser particles.

Equipment for the study of ultrafine powders by positron annihilation techniques has been designed in the Department and is currently being constructed.

4.5. Brazing of New Advanced Materials

The EEC-BRITE supported research programme on the development of improved new Ni-Cr-based brazing filler metals was continued in collaboration with the partners from FRG and the Netherlands. Extensive test programmes determine the wetting-, tensile- and fatigue (10^4-10^8 cycles) properties. Also, the corrosion resistance in a diluted HNO_3 solution at 75°C has been investigated on joints brazed with four selected commercial filler metals - one from each of the types - P, Si, B and Si plus B. The effect of the compositional changes of the new experimental alloys will be evaluated. The influence of the mechanical and corrosion properties of the brittle phases initially formed is detected in a narrow gap without intermetallics and in a wider gap (100 μm) containing continuous intermetallics. So far SEM and TEM investigations have been carried out on the various phases present in the wider gap of the specimens best able to resist corrosion to find the relations between these phases and the details of corrosion attack. Based on these results, new experimental filler-metals have been compared and produced.

Micrograph of brazed joint using NiBSi filler material.

In the Danish Center for Advanced Technical Ceramics the joining programme was continued. Brazing of PS ZrO_2 (3% Y_2O_3) was investigated using a selected active brazing filler metal (70 Ag, 27Cu, 3 Ti) based on our earlier reported results on the joining of Al_2O_3 to Al_2O_3 with Ti-containing active Ag Cu filler metals. A maximum 4 point bend strength of 300 MPa was obtained on unpolished PS ZrO_2 specimens brazed at 900°C. The fractures were located very near to a high containing 0,5 - 1 μm thick layer formed at the interface between the filler metal and the ZrO_2. SEM and TEM investigations are carried out to analyse the formed intermediate phases. Diffusion joining of Al_2O_3 and PS ZrO_2 was also started. Several components involving a ZrO_2 tube joined between a Al_2O_3 lid and a Al_2O_3-tube using PZ foils were fabricated with good reproducibility concerning tightness and thermal cycling. The effect of the diffusion joining parameters - time, temperature and pressure - will be investigated when the more elaborate diffusion joining equipment is completed. Initial investigations on ceramic/metal joints will also start.

In the Danish Center for Powder Metallurgy a joining programme has been launched. So far a literature survey has revealed that very little has been published in this area on sintered products based on steel - and stainless steel powder - especially not on sintered and finished materials.

Contract work was continued on industrial applications of dip brazing of aluminium alloys as well as vacuum brazing of stainless steels, superalloys, ceramics and ceramic/metal.

5. DANISH AND INTERNATIONAL COLLABORATION

The Department is engaged in the following types of international collaboration: joint scientific and technical projects, committee work, reception of research fellows, and technical and scientific meetings. The Department was represented in the following committees:

International

The Information Exchange Group under the European Space Agency on Carbon Fibre Reinforced Plastics,

The Halden Programme Group, OECD,

The IRDAC Working Party No. 6 on Materials Research and Development (EEC),

The IAEA International Working Group on »Water Reactor Fuel Performance and Technology« (Chairmanship),

The Permanent Comm e for Stress Analysis,

The Project Committee of the Battelle High Burnup Performance Programme (HBEP),

The European Group on Fracture (EGF),

The EEC Working Group on »Reference Materials for the Elemental Analysis of Plutonium«,

The COST 501 Management Committee on Materials for Energy Conversion Using Fossil Fuels,

The European Coal and Steel Community, Executive Committee No. 5: Failure Mechanisms and Design,

The Euratom Neutron Radiography Working Group (Chairmanship),

The Nordic Committee for Thermal Analysis (Chairmanship),

The Technical Commission of the International Institute of Welding, Commission I, »Gas Welding and Allied Processes«.

The Fusion Technology Steering Committee,

The Fusion Materials Expert Groups of the European Fusion Technology Programme: »Structural Materials« and »Plasma Facing Materials«,

The Editorial Board of »Composites Science and Technology«, »Journal of Materials Education«, »Thermochimica Acta«, »Textures and Microstructures« and »Journal of Nuclear Materials«,

The Nuclear Corrosion Working Party under the European Federation of Corrosion,

The ISPRA Cyclotron Users Committee,

International Advisory Committee for the International Conference on Fusion Reactor Materials,

The International Advisory Committee on »International Conferences on Positron Annihilation«.

Danish

The Executive Committee of the Danish Metallurgical Society,

The Research Committee of the Danish Engineering Association (Chairmanship),

The Board of Governors of Risø (academic and general staff representatives),

The Danish Ministry of Energy; Advisory Group for Fuel Cells, Advisory Group for Transport, Steering Committee for Danish Solid Oxide Fuel Cells Programme (Project Manager),

The Danish Materials Technology Development Programme; Centre of Advanced Technical Ceramics (Director), Centre of Powder Metallurgy (Director), Centre of Polymer Composites (Project Manager).

6. EDUCATION AND TRAINING

Niels Hansen and Kaj Rørbo gave regular lectures on materials science to undergraduates at the Danish Academy of Engineering. As part of the COMETT course on »Fibre Reinforced Materials«, lectures were given by Svend Ib Andersen, Povl Brøndsted, Bjørn Johansen, Hans Lilholt, and Aage Lystrup. The following acted as external examiners: Torben Leffers at the University of Newcastle, Australia and at the Technical University of Denmark; Dorte Juul Jensen at the Technical University of Norway; Ole Toft Sørensen at the Technical University of Denmark. Ole Bøcker Pedersen was a visiting scientist at the University of Cambridge, U.K. Andy Horsewell was a guest professor at EPFL, Lausanne, Switzerland.

Post-Graduate Projects

The following students carried out research work within the Department towards their Ph.D. degrees at Danish universities:

Charlotte Clausen:	Technical University of Denmark/Danish Research Academy, »Characterization of Interfaces in Ceramics by Electron Microscopy«.
Peter L. Husum:	Technical University of Denmark, »The Fabrication and Characterization of Advanced Technical Ceramics«.
Ole Jørgensen:	Technical University of Denmark/Danish Research Academy, »Impact Damage in Polymer Composites«.
Bo Jeppesen:	Technical University of Denmark, »Finite Element Theory for Thick Laminated Plates with Damage Development«.
Torben Lorentzen:	Aalborg University Centre, »Measurement of Internal/Residual Stresses by Neutron Diffraction«.
Bent Sørensen:	Technical University of Denmark/Danish Research Academy, »Fibre-Reinforced Ceramics«.
Niels Jacob Sørensen:	Technical University of Denmark/Danish Research Academy, »Thermomechanical Properties of Metal Matrix Composites at High Strains and Temperatures«.

The following students from universities outside Denmark worked towards the Ph.D. within the Department:

Ian Barker:	Brunel University, UK, »Development of Techniques to Measure Local Misorientations and Application of Such Techniques in the Study of Deformed and Partly Recrystallized Structure in Metals«.
David Tricker:	University of Cambridge, UK, »The Electron Microscopical and Electrical Characterization of Boundaries in Solid Oxide Fuel Cell Materials«.
Phil Withers:	University of Cambridge, UK, »Interfaces and Internal Stresses in Metal Fibre Composites«.

7. ECONOMY

The Department receives its financial support partly through basic governmental funding, and partly through national and international project oriented funds.

The numbers given are in units of 1000 Danish kr. In parantheses the equivalent number in units of 1000 US$ (100 DKR. equals 13.5 US$) is given.

Income

	DKR. x 1000	$ x 100
Basic funding: (Ministry of Energy)	21100	2900
Project funding:	18600	2500
	39700	5400

Investments

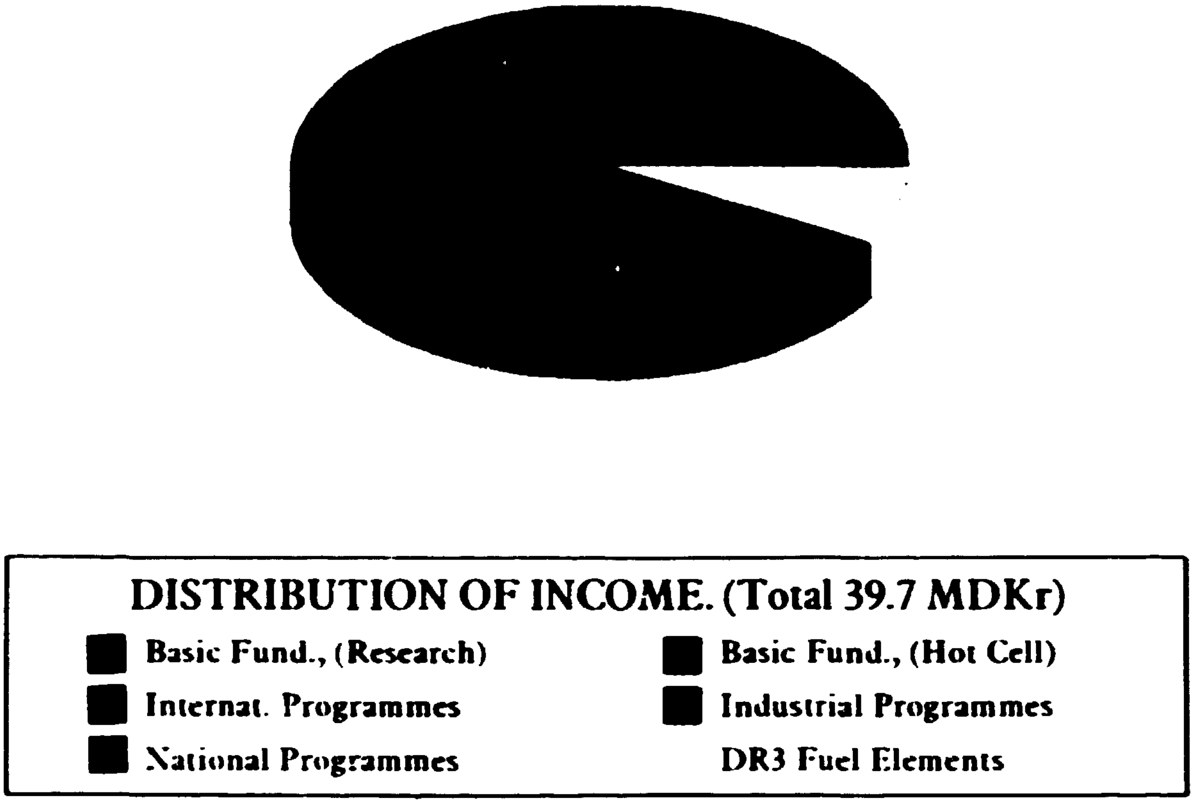
	1000 DKR.	1000 US$
Equipment	4700	650
Financing:		
Basic Funds	700	95
Project Funds	4000	555

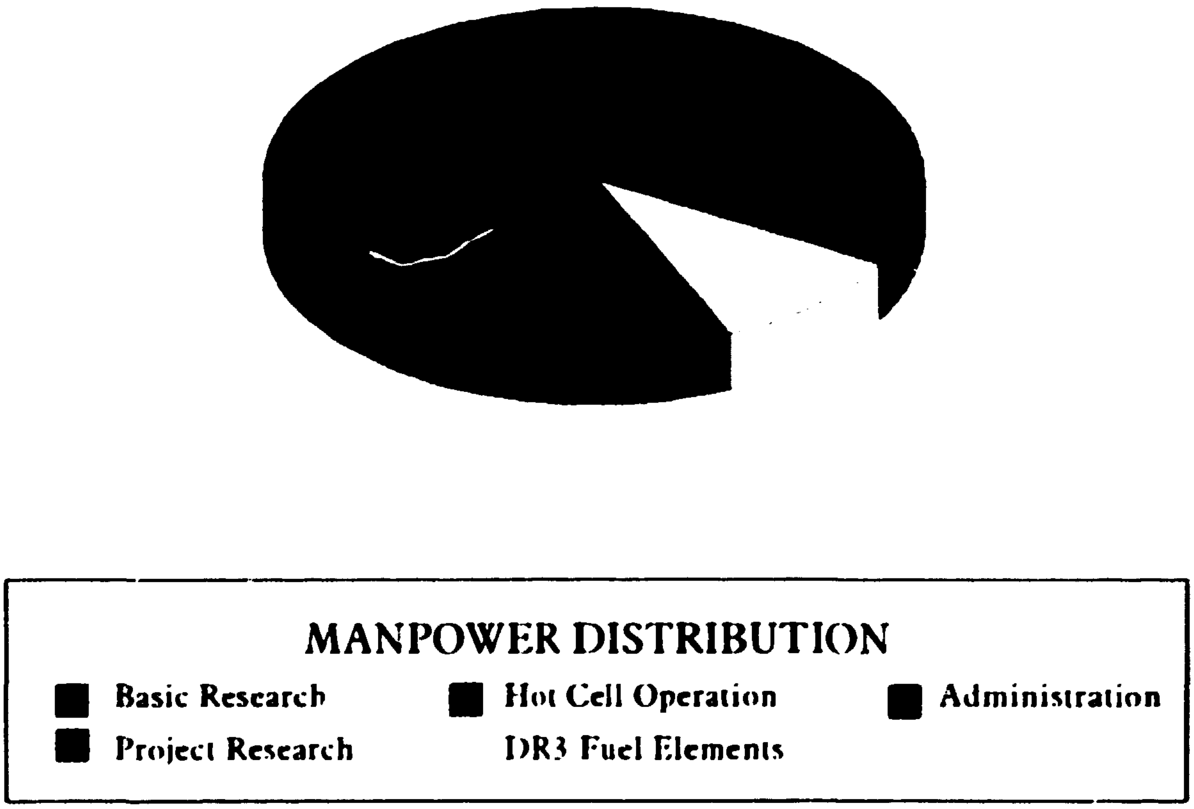
Expenditure 1989

	Basic Funds		Project Funds		Total	
	1000 DKR.	1000 US$	1000 DKR.	1000 US$	1000 DKR.	1000 US$
Salary	16600	2250	7200	970	23.800	3220
Running expenses:	3800	510	4000	540	7800	1050
Equipment:	700	90	3200	300	3900	390
Tax:	-	-	500	70	500	70
Administrative charges:	-	-	2600	350	2600	350
Total:	21100	2850	17500	2230	38600	5080

Distribution of Staff on Research Topics (31 December 1989)

Topic	Scientific Staff	Technical Staff
Materials Science	9	5
Materials Engineering	11	10
Materials Technology	13	16
Hot Cell Operation	1	12
Administration	2	7
Total:	36	50
Graduates	4	-
Apprentices	-	8


DISTRIBUTION OF INCOME. (Total 39.7 MDKr)
Basic Fund., (Research)
Basic Fund., (Hot Cell)
Internat. Programmes
Industrial Programmes
National Programmes
DR3 Fuel Elements


MANPOWER DISTRIBUTION
Basic Research
Hot Cell Operation
Administration
Project Research
DR3 Fuel Elements

8. STAFF OF THE DEPARTMENT

Head of Department
Niels Hansen

Scientific Staff
Adolph, Eivind
Ananthan, V.S. 2)
Andersen, Svend I.
Bagger, Carsten
Bentzen, Janet
Bilde-Sørensen, Jørgen B.
Borring, Jan
Borum, Kaj
Brøndsted, Povl
Bøcker Pedersen, Ole
Carlsen, Hans
Christensen, Jørgen
Clausen, Charlotte 4) 1)
Debel, Christian P.
Domanus, Josef
Eldrup, Morten
Gotthjælp, Klaus
Gundtoft, Hans E.
Horsewell, Andy
Johansen, Bjørn S.
Juul Jensen, Dorte
Jørgensen, Ole 1) 4)
Knudsen, Per
Leffers, Torben
Lilholt, Hans
Liu, Yi-Lin 3)
Lorentzen, Torben 5)
Lystrup, Aage
Løgstrup Andersen, Tom
Malmgren-Hansen, Bjørn
Mogensen, Mogens
Nyberg, Eric 6)
Poulsen, Finn W.
Rørbo, Kaj
Schrøder Pedersen, Allan
Singh, Bachu N.
Sørensen, Bent 1) 4)
Sørensen, Niels J. 1) 4)
Tetzschner, Mogens
Toft, Palle
Toft Sørensen, Ole
Toftegaard, Helmuth
Waagepetersen, Gaston

Technical Staff
Adrian, Frank
Andersen, Axel B.
Aukdal, Jørgen
Borchsenius, Jens
Bülow-Christensen, Carl J.
Bräuner, Carsten
Christensen, Svend E.
Cooper, Dennis
Dreves Nielsen, Poul
Frederiksen, Henning
Friedrichsen, Uwe J.
Hersbøll, Bent
Hjort Petersen, Jan
Jensen, Finn
Jensen, Knud
Jensen, Palle V.
Jespersen, John
Kjøller, John
Klitholm, Cliver
Larsen, Bent
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Ove
Nielsen, Palle
Nilsson, Helmer
Nørregård Gravesen, Niels
Olesen, Preben B.
Olsen, Benny F.
Olsen, Bent
Olsen, Henning
Olsen, Ole
Olsson, Jens
Paulsen, Henrik
Pedersen, Børge
Pedersen, Knud E.
Pedersen, Niels J.
Robl, Steen
Sandsted, Kjeld
Strauss, Torben
Sørensen, Erling
Thomsen, Leif
Aagesen, Sven

Office Staff
Dreves Nielsen, Elsa
Helbo Pedersen, Anne
Hoffmann Nielsen, Lis
Jørgensen, Lone
Lauritsen, Grethe W.
Sørensen, Eva
Thomsen, Ann

Apprentices
Christensen, Kim
Enghave, Christian
Jacobsen, Mogens
Jensen, Kjeld L.
Jensen, Lars
Madsen, Niels O.
Olsen, Morten
Westergaard, Kenneth

1) Post-graduate student from the Technical University of Denmark.
2) Visiting scientist from the University of Newcastle, Australia.
3) Visiting scientist from Jiangsu Aeronautical Company, China.
4) Post-graduate student from the Danish Research Academy.
5) Post-graduate student from Aalborg University Centre, Denmark.
6) Visiting scientist from Washington State University, U.S.A.

Four academic staff members joined the Department and two left during 1989.
One technical staff member left the Department during 1989.

9. VISITING SCIENTISTS AND STUDENTS

The following guest scientists worked in the Department during 1989:

Dr. M. El Sayed Ali, Atomic Energy Centre, Cairo, Egypt, 2 - 20 June.
Dr. V.S. Ananthan, University of Newcastle, Australia, 1 Jan. - 31 Dec.
Dr. Claire Barlow, University of Cambridge, United Kingdom, 1 - 29 Sep.
Dr. Charles E. Bream, Consultant, Cruckmeole House, United Kingdom, 15 Jul - 15 Oct.
Dr. Adam Bunsch, Institute of Metallurgy, Krakow, Poland, 1 - 31 Dec.
Dr. Samia El-Houte, Atomic Energy Centre, Cairo, Egypt, 2 - 20 Jun.
Dr. Alan Foreman, Harwell Laboratory, United Kingdom, 9 - 30 May.
Dr. David S. Gelles, Battelle Pacific Northwest, WA, U.S.A., 15 Jun - 15 Aug.
Dr. Darcy A. Hughes, Sandia National Laboratory, CA, U.S.A., 1 - 30 Apr.
Dr. M. Hartmanova, Institute of Physics, Bratislava, Czecheslovakia, 1 - 14 Jun.
Dr. Bruno Kindl, National Research Council, Canada, 15 Mar - 15 Jun.
Professor Meng Guang-Yao, University of Science and Technology of China, China, 1 Jul - 1 Oct.
Dr. Vaclav Novak, Institute of Physics, Czechoslovak Academy of Sciences, Prague, Czechoslovakia. 3 - 15 Sep.
Eric Nyberg, Washington State University, WA, U.S.A., 16 Oct - 31 Dec.
Dr. Peng Ding-kun, University of Science and Technology of China, China, 1 Jul - 1 Oct.
Professor Brian Ralph, Brunel University, United Kingdom, 15 - 19 Mar.
Dr. H. Schramm, Technical University of Delft, Netherlands, 14 - 21 Aug.
Dr. Jim F. Stubbins, University of Illinois, U.S.A., 31 May - 6 Jul.
Dr. Helmuth Trinkaus, KFA Jülich, West Germany, 30 Jan - 2 Feb.
Professor Wang Hong, University of Science and Technology of China, China, 1 Jan - 1 May.
Dr. Chung H. Woo, Atomic Energy of Canada, Canada, 5 - 15 Oct.
Dr. Yi-lin Liu, Jiangsu Aeronautical Company, Peoples Republic of China, 1 Jan - 31 Dec.

The following students worked in the Department during 1989:

John Barker, Northwestern University, ILL., U.S.A., 26 - 31 Dec.
Naijin Fei, Technical University of Denmark, 1 Jan - 30 May.
B.C. Diederik Jaspers, Technical University of Delft, Netherlands, 1 Aug - 31 Oct.
Maarten Joost de Mol van Otterloo, Technical University of Delft, Netherlands, 1 Aug - 31 Oct.
Daniele Mari, EPFL Lausanne, Switzerland, 15 - 24 Mar and 9 Nov - 4 Dec.
Rao Ningling, University of Science and Technology of China, China, 1 Sep - 31 Dec.
Frank L. Ross, Colombia University, N.Y., U.S.A., 22 May - 14 Jul.
Yin Man Tang, Brunel University, United Kingdom, 23 Mar - 30 Aug.
Yu Shu, Beijing Research Institute, China, 1 Jan - 30 Apr.

PUBLICATIONS

1. Als-Nielsen, J.; Andersen, N.H.; Clausen, K.N.; Michelsen, P.; **Poulsen, F.W.**, Experiments on Palladium- and Titanium-Deuterium Systems with Reference to Studies on Cold Fusion. Risø-M-2806 (1989) 12 p.

2. **Ananthan, V.S.**; Hall, E.O., Shear and Kink Angles at the Lüders Band Front. Scr. Metal. (1989) v. 23 p. 1075-1078.

3. **Andersen, S.I.; Brøndsted, P.; Adrian, F.**, Cyclic Thermomechanical Behaviour of Steel. I: New Materials and Processes. Proceedings. 5. Scandinavian Symposium on Materials Science, Copenhagen, 22-25 May 1989. Hansson, I.L.H.; Lilholt, H. (eds.), (Danish Society for Materials Testing and Research, Copenhagen, 1989) p. 89-102.

4. **Andersen, S.I.; Lilholt, H.**, Revnedannelse ved langtidsudmattelse af glasfiber/polyester kompositmaterialer. I: Nedbrydning af materialer: Korrosion, slid, revner. (Crack Formation during Long-Term Fatigue of Glass-Fibre/ Polyester Composites. In: Degradation of Materials: Corrosion, Wear and Cracks). (In Danish). Dansk Metallurgisk Selskab. Vintermøde, Fåborg, 4-6 Jan 1989. Lilholt, H.; Gundel, P.H. (eds.), (Dansk Metallurgisk Selskab, Lyngby, 1989) p. 19-39.

5. **Andersen, S.I.; Lilholt, H.**, Stiffness Changes during Fatigue of Angle-ply Glass/Polyester of high Quality under very large Number of Cycles. I: Developments in the Science and Technology of Composite Materials. 3. European Conference on Composite Materials. ECCM-3, Bordeaux, 20-23 Mar 1989. Bunsell, A.R.; Lamicq, P.; Massiah, A. (eds.), (Elsevier Applied Science, London, 1989) p. 529-534.

6. **Bagger, C.**, Management of Huge Amounts of Pie Data. I: Symposium on Post Irradiation Examination in Nuclear Programme. Vol. 1: Invited Talks. Post Irradiation Examination in Nuclear Programme, Bombay, 29 Nov - 1 Dec 1989. (Bhabha Atomic Research Centre, Bombay, 1989) 7 p.

7. **Bagger, C.; Johansen, B.S.**, Refabrication of Irradiated Nuclear Fuel Rods. I: Symposium on Post Irradiation Examination in Nuclear Programme. Vol. 1: Invited Talks. Post Irradiation Examination in Nuclear Programme, Bombay, 29 Nov - 1 Dec 1989. (Bhabha Atomic Research Centre, Bombay, 1989) 11 p.

8. **Bagger, C.; Mogensen, M.; Knudsen, P.**, Unique Techniques to Investigate High Burn-up Fuel. Nucl. Europe (1989) v. 8 (no.1/2) p. 17.

9. Barker, I.; **Hansen, N.**; Ralph, B., The Development of Deformation Substructures in Face-Centred Cubic Metals. Mater. Sci. Eng. A (1989) v. 113 p. 449-454.

10. Barlow, C.Y.; **Hansen, N.**, Deformation Structures in Aluminium Containing Small Particles. Acta Metall. (1989) v. 37 p. 1313-1320.

11. Barlow, C.Y., Stradivarius' Secret - Was it All in the Varnish? Risø Nyt (1989) v. 2 p. 13-15.

12. Bay, B.; **Hansen, N.**; Kuhlmann-Wilsdorf, D., Deformation Structures in Lightly Rolled Pure Aluminium. Mater. Sci. Eng. A (1989) v. 113 p.385-397.

13. **Bentzen, J.J.; Bilde-Sørensen, J.B.**; Kindl, B.; **Paulsen, H.; Poulsen, F.W.**, Thin Tape Cast Zirconia for Solid Oxide Fuel Cells. I: New Materials and Processes. Proceedings. 5. Scandinavian Symposium on Materials Science, Copenhagen, 22-25 May 1989. Hansson, I.L.H.; Lilholt, H. (eds.), (Danish Society for Materials Testing and Research, Copenhagen, 1989) p. 149-157.

14. **Bilde-Sørensen, J.B.; Hansen, N.; Juul Jensen, D.; Leffers, T.; Lilholt, H.; Pedersen, O.B.** (eds.), Materials Architecture. 10. Risø International Symposium on Metallurgy and Materials Science, Risø, 4-8 Sep 1989. (Risø National Laboratory, Roskilde, 1989) 688 p.

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35.-36. **Pedersen, O.B.**, Mapping and Modelling Work-Hardening and Fatigue. Presented at Department of Materials Science and Engineering, University of Surrey, Guildford, 10 Nov 1989; Condensed Matter and Material Colloquium, Los Alamos National Laboratory, New Mexico, USA, 21 Dec 1989.

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48. **Singh, B.N.**, Helium Diffusion Through Crystal Lattice, Dislocations and Grain Boundaries. Presented at Symposium on Noble Gases in Metals: TMS/AIME Annual Meeting, Las Vegas, 28 Feb to 2 March 1989.

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POSTERS

1. **Bentzen, J.J.**; Schwartzbach, H., Electrical Conductivity, Structure, and Thermal Expansion Behaviour of ZrO_2-CeO_2-Gd_2O_3-Y_2O_3 Solid Solutions. Presented at 7. International Conference on Solid State Ionics, Hakone, Japan, 5-11 Nov 1989. To be published in Solid State Ionics 1990.

2. Heinisch, H.L.; **Singh, B.N.**, The Morphology of Collision Cascades as a Function of Recoil Energy. Presented at 4. International Conference on Fusion Reactor Materials. Kyoto, Japan, 4-8 Dec 1989. To be published in J. Nucl. Mater.

3. **Horsewell, A.**; **Singh, B.N.**; Sommer, W.; Heinisch, H.L., Defect Structures in Copper Irradiated with Fast Neutrons, Spallation Neutrons, 14 MeV Neutrons and 600-800 MeV Protons. Presented at International Conference on Fusion Reactor Materials-4, Kyoto, Japan, 4-8 Dec 1989. To be published in J. Nucl. Materials.

4. **Lauritsen, G.W.**, Risø National Laboratory. Presented at GASAT 5, Gender and Science and Technology, 5. International Conference 1989, Technion - Israel Institute of Technology, Haifa, Israel, 17-22 Sep 1989. To be published.

5. **Singh, B.N.**; Foreman, A.J.E., Some Limitations of Simulation Studies using ppm to dpa Ratio as Helium Generation Rate. Presented at 4. International Conference on Fusion Reactor Materials, Kyoto, Japan, 4-8 Dec 1989. To be published in J. Nucl. Mater.

6. **Toft Sørensen, O.**; **Strauss, T.**, Wear Resistant Zirzonia Toughened Ceramics. Presented at Ceramic Materials '89, Gothenburg, Sweden, 5-7 Jun 1989.

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10. Woo, C.H.; **Singh, B.N.**; Heinisch, H.L., Effects of Collision Cascades on Recombination and Clustering of Point Defects. Presented at 4. International Conference on Fusion Reactor Materials, Kyoto, Japan, 4-8 Dec. To be published in J. Nucl. Mater.

Risø National Laboratory is a broad-based research organization with primary research activities in energy, the environment and in materials. There is a total of 930 employees.

The Metallurgy Department* employs 45 scientists and engineers and 50 technical staff. The research programmes include basic studies of materials structure and properties, structural mechanics and materials testing, and processing techniques for polymer composites, powder metallurgical products and engineering ceramics. Advanced characterization techniques used in the Department are electron microscopy, positron annihilation, neutron diffraction, small angle neutron scattering and mechanical testing.

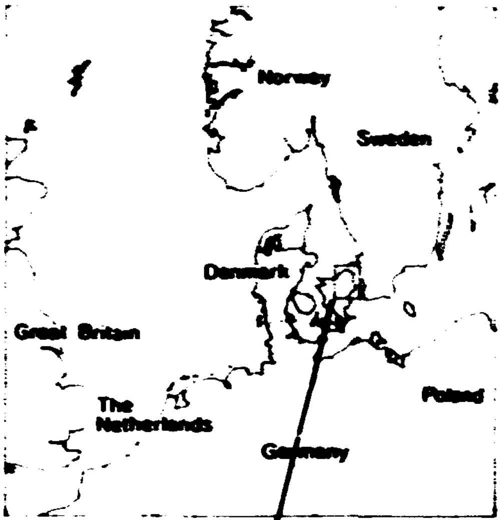
The activities of the Metallurgy Department are supported by a combination of basic government funding, national, EEC and international project funded research and through industrial contracts.

Within the Danish Programme for Materials Technology, the Metallurgy Department participates in the Centres of Polymer Composites, Advanced Technical Ceramics and Powder Metallurgy.

*The department has changed its name to the "Materials Department" as of May, 1990.

Materials Department
Risø National Laboratory
DK-4000 ROSKILDE
DENMARK

TEL. + 45 42 371212
FAX + 45 42 351173



Risø is located in pleasant surroundings
35 km west of Copenhagen,
6 km north of Roskilde.